1982

Energy Analysis of Small-scale Ethanol Distillation

Scott Stampe

ENERGY ANALYSIS OF SMALL-SCALE

ETHANOL DISTILLATION

BY

SCOTT STAMPE

A thesis submitted
in partial fulfillment of the requirements for the
degree of Master of Science
Major in Agricultural Engineering
South Dakota State University
1982

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This thesis is approved as a creditable and independent investigation by a candidate for the degree, Master of Science, and is acceptable for meeting the thesis requirements for this degree. Acceptance of this thesis does not imply that the conclusions reached by the candidate are necessarily the conclusions of the major department.

Darrell W. De Boer — Date
Thesis Adviser

Dennis L. Moe — Date
Head of Major Department

ACKNOWLEDGEMENTS

I wish to extend my appreciation first and foremost to Dr. Tom S. Chisholm for insight and guidance in the design of the experimentation described herein. I have gained the deepest respect for and confidence in him; first as a fine person, and then as a fine engineer.

I wish to thank Dr. Darrell W. De Boer for his patience and persistence in completing the final draft of this thesis. Dr. William L. Tucker deserves credit for much of the work on the statistical analyses presented. Credit for the photography goes to a fellow graduate student and friend, Mr. Peter H. Leiferman.

Lastly, I wish to extend my tenderest appreciation to the force which has been most responsible for providing both the atmosphere and motivation necessary to complete this work. Thank you, Angela, for your love and understanding throughout this effort.

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ABSTRACT

Steam consumption associated with the small-scale fractional distillation of ethanol at the South Dakota State University Alcohol Fuels Research Facility has been determined. During 1981, seven, ten, and thirteen percent ethanol concentrations in corn-beer were distilled to a 92.5 percent fuel ethanol product. Residual ethanol concentrations in spent stillage were studied at average levels of 0.13, 0.15, and 0.30 percent by volume. The total system energy requirement averaged 3.88 megajoules per liter (13,900 Btu per gallon) of anhydrous-equivalent ethanol, exclusive of boiler efficiency.

Emperical relationships were established between the distillation energy requirement and two operating parameters. These parameters were studied in two independent experiments. In experiment one, "center stripper temperature" served as an indicator of the parameter "stillage residual ethanol concentration." The former demonstrated an inverse, linear relationship (R^2 = 0.88) with the energy requirement of distillation when combined with the variables "center rectifier temperature" and "product flow rate." In the second experiment, the parameter "corn-beer ethanol concentration" demonstrated an inverse, quadratic relationship (R^2 = 0.99) with the energy requirement of distillation.

The total steam energy requirement was partitioned into components. Heating of the corn-beer required 20 percent; relux generation, 38 percent; product condensation, 18 percent; and system thermal losses absorbed the remaining 24 percent of total energy consumption.

INTRODUCTION

The production of ethyl alcohol from farm grain products for use as a liquid fuel is an exciting, yet controversial concept. There are potential advantages as well as disadvantages for the farmer, or nation, which can simultaneously guarantee a renewable supply of energy and a market for farm produce.

The conversion of corn, or other starch crops, to ethanol is beneficial to the United States in a number of ways. Energy that is produced within the United States reduces the import of foreign crude oil and thus the federal deficit. An alcohol fuel industry creates jobs and therefore adds to the tax base of the national economy. Such an industry can provide a market that could stabilize grain prices. During the ethanol production process only the starch is removed from the grain. This leaves 100 percent of the protein for use as human food or animal feed.

There are also several disadvantages for using ethanol as a liquid fuel. The current cost for producing ethanol is greater than the cost of the gasoline it replaces. Therefore, a substantial economic subsidy for fuel ethanol is required to make it competitive in the marketplace. Also, in general, a higher grain market dictates higher consumer food prices.

Another disadvantage associated with fuel ethanol production is the complexity of the production process. The technology for converting grain to ethanol has been known for decades, as evidenced by writings of Brachvogel (1907), Herman et al. (1942), and Underkofler and Hickey (1954). However, it requires significant engineering and microbiological expertise. This author has the opinion that the production of fuel ethanol is largely out of the hands of the masses and into the hands of a relatively few companies that have the necessary technology.

There is also a physical limitation to the use of fuel ethanol made from corn. According to the United States Bureau of the Census (1980), in 1978 the United States produced 0.18 billion metric tons (seven billion bushels) of corn for grain. Even if the impossible event of total conversion to ethanol could occur, only 68 billion liters (18 billion gallons) of ethanol would be produced. In 1978, we consumed 470 billion liters (125 billion gallons) of gasoline.

The place of fuel ethanol in American's energy scene lies somewhere between being irrelevant and being a panacea. Current thought lies in the more economical use of ethanol as an octane booster rather than as a substantial fuel replacement. The full potential and feasibility of the concept depends upon considerations in no less than three areas; those of technical, economic, and political arenas.

It is the intent of this thesis to focus on one facet of the technical aspect of ethanol production. The emphasis lies with the examination of the magnitude and makeup of the steam consumption of the typical distillation process that separates ethanol from a corn and water slurry. The evaluations are based upon operation of a small, commercial, 30.5 centimeter (12.0 inch) diameter distillation tower at the South Dakota State University Alcohol Fuels Laboratory during 1981. A brief history of the establishment and purpose of the fuel alcohol project is presented as Appendix A.

OBJECTIVES

The general objective of this research was to evaluate the small-scale, fractional distillation of corn-beer to a 92.5 percent ethanol by volume product. Distillation was one portion of the dry-milling process of fuel alcohol production employed at South Dakota State University.

Specific objectives of this research are:

1. To establish steam energy requirements for the distillation of corn-water-ethanol slurries by studying various stillage residual ethanol concentrations.
2. To establish steam energy requirements for the distillation of corn-water-ethanol slurries by studying various corn-beer ethanol concentrations.
3. To study the interrelationships among 22 distillation system variables.
4. To partition the distillation tower steam consumption into four components: corn-beer heating, product condensation, reflux generation, and system thermal losses.
5. To evaluate the distillation equipment and accessories, data collection procedures, and methods of laboratory analysis.

LITERATURE REVIEW

The concept of employing ethanol as a motor fuel has periodically surfaced since the invention of the internal combustion engine. The idea that ethanol production can make farmers energy producers appealed to a farm boy from Dearborn, Michigan, named Henry Ford. In the 1880's, he designed one of his earliest automobiles, the quadricycle, to burn ethanol (Bossong, 1980).

"Despite the intense competition from gasoline, alcohol fuels were used to power American cars well into the 1920's and 1930's," continues Bossong (1980). During World War II, a surge in the production of ethanol helped to produce increased amounts of synthetic rubber necessary to run this country's war machine. Following the war, the decreased need for synthetic rubber and the availability of cheap petroleum created an enormous slump in the production of industrial ethanol.

Very little ethanol was used to power this country's post war automobiles until 1973 when Nebraska passed a three cent per gallon cut in state fuel taxes. This applied only for fuel containing a minimum of ten percent agricultural ethanol of at least 190 proof, that is, 95 percent ethanol by volume. The present state of fuel ethanol production has grown out of this attempt to replace foreign oil with domestically produced fuel.

The technology employed for the production of ethanol from agricultural crops is as old as ethanol's use as a fuel. According to

Calingaert (1925), a Frenchman named Barbet designed a successful plant-scale fractional distillation process in 1890. High pressure steam was almost universally employed for the cooking of grain as far back as 1907 (Brachvogel, 1907). By 1942, continuous pressure cooking was described by Herman, et al. (1942), as the latest in modern distillery technology.

The French have long been considered the fathers of distillation technology. Mariller (1945) presented two intricate prediction equations which emperically estimate the steam energy requirements for distillation. Variables employed in the relationships include the concentration of ethanol in the beer, the required quantity of reflux, and the ethanol concentration of the final product. Mariller also cited longstanding French methodology in measuring energy consumption of distillation. He presented eight sets of data which define energy requirements for the distillation process that was typically employed in France.

A desire to reduce energy expenditures resulted in new approaches to distillation. Calingaert (1925) and Reich (1929) indicated that, during this period, the minimum amount of energy required to distill a liter of 95 percent by volume ethanol product in large alcohol plants was about 9.5 megajoules (34,000 Btu per gallon of ethanol). It was not uncommon to find plants requiring 15 to 16 megajoules per liter (53,000 to 58,000 Btu per gallon) of product (Freshwater, 1951; Campion 1952). Freshwater (1951) cited Mariller in 1913, and Reich, in 1938, as suggesting a combined system of evaporation of spent stillage and distillation. Campion (1952) made reference to Barbet, who used steam in a double effect arrangement. Heat available from one column is used a second time to heat a second column operating at a reduced pressure. Another possibility presented was to use the vapors of the rectifying column to heat the stripping column. This can be done if the latter is operated at a reduced pressure. Freshwater's paper suggests additional multiple effect arrangements, as well as proposing vapor recompression by use of a heat pump.

These innovations provided industrial alchohol production with the methods for reducing energy expenditures by 50 percent. Campion (1952) predicts a 48 percent energy savings while Dobson (1950) reports an energy expenditure in a two-column still of 4.9 megajoules per liter (17,500 Btu per gallon) of ethanol. A reduction in the energy expenditure of distillation of ethyl alcohol has certainly been accomplished at the large, commercial distilleries.

The purpose of these distilleries has been the production of a potable beverage or an industrial grade product. The energy required to distill ethanol for use as fuel can be less than that for beverage or industrial purposes because the extra equipment employed in commercial distilleries is not required.

Jantzen and McKinnon (1980) made a preliminary study of small-scale, fuel ethanol facility in Campo, Colorado. They found that 4.49 megajoules per liter (16,100 Btu per gallon) of ethanol in the form of steam energy was required to distill a 95 percent ethanol product from a 12 percent corn-beer. This figure was for an atmospheric, single effect, two-column unit and included energy loss due to boiler efficiency.

A small-scale fuel ethanol plant in Cuba was examined by Aries

(1947). The distillation unit was a two-column unit capable of producing a 96 percent ethanol by volume product. Table 1 presents Aries' results as the beer ethanol concentration and reflux ratio were varied. The reflux ratio is an operating parameter of a distillation column defined as the ratio of the quantity of liquid ethanol returned to the top of the rectifier column to the quantity of ethanol product removed from the distillation process. The reflux ratio influences the concentration of ethanol in the product. However, for large ratios (greater than four to one), the ratio can be employed as a measure of the efficiency of the distillation unit. Steam consumption ranged from 6.3 megajoules per liter (22,700 Btu per gallon) to 14.2 megajoules per liter (51,000 Btu per gallon) of product, respectively.

Table 2 presents the effects of varying inlet beer temperatures upon the steam consumption of the same distillation unit. Calculations can show that the increase in steam energy matches that needed to heat the inlet beer to its boiling point.

Further research is needed to determine the effects of other parameters on the steam requirements of distillation.

Table 1: Steam Consumption, in Megajoules Per Liter of 96 Percent Alcohol, Corresponding to Different Percentages of Alcohol in Beer, and Different Reflux Ratios (Aries, 1947)

Percent Alcohol in Beer	Reflux Ratio 5/1	6/1	7/1	8/1	9/1	10/1	11/1	12/1	13/1
	megajoules per liter ethanol								
4.0	8.2	8.9	9.7	10.4	11.2	12.0	12.7	13.5	14.2
4.5	7.8	8.5	9.3	10.0	10.8	11.5	12.3	13.1	13.8
5.0	7.4	8.2	8.9	9.7	10.5	11.2	12.0	12.7	13.5
5.5	7.2	7.9	8.7	9.4	10.2	10.9	11.7	12.5	13.2
6.0	6.9	7.7	8.5	9.2	10.0	10.7	11.5	12.2	13.0
6.5	6.8	7.5	8.3	9.0	9.8	10.5	11.3	12.1	12.8
7.0	6.6	7.4	8.1	8.9	9.6	10.4	11.1	11.9	12.7
7.5	6.5	7.2	8.0	8.7	9.5	10.2	11.0	11.8	12.5
8.0	6.3	7.1	7.8	8.6	9.4	10.1	10.9	11.6	12.4

Table 2: Variations in the Steam Consumption of a Two-Column Distillation Unit, Operating Under 6/1 Reflux and 6.5 Percent Beer, With Different Inlet Temperatures of the Beer (Aries, 1947)

Temperature, ^{o}C.......:	38	43	49	54	60	66	71	77	82	88	94
Energy Per Liter, MJ/l:	10.6	10.2	9.9	9.6	9.2	8.9	8.5	8.2	7.9	7.5	7.2

ETHANOL PRODUCTION PROCESS

The physical nature as well as the composition of the fluid requiring distillation, referred to as corn-beer, should be defined to characterize the ethanol-water separation process. The ethanol production process employed at South Dakota State University used a batch cook and fermentation procedure followed by continuous flow distillation. In a batch process, each tank is independently filled and time is allowed for the reaction within to be completed. In a continuous flow system, each step of the process proceeds concurrently. Refer to the time-temperature diagram of Figure B-1 in Appendix B for a representation of the whole process. Cook-fermentation tanks are staggered so as to provide a steady flow of corn-beer to the distillation tower. Although continuous flow centrifugation of the spent stillage following distillation is employed to remove 70 to 80 percent of the residual corn solids, none of the clarified fluid is recirculated into the following batch. Use of a portion of this waste is recommended in a commercial operation to extract additional ethanol from the corn. However, an uncontrolled variable is thereby possibly admitted into the experimentation. Increasing the concentration of suspended solids, especially fats, could increase extraneous variation in the observed data.

Milling, cooking, and fermentation procedures are now described.

Milling

Locally produced, number two, yellow corn undergoes a dry milling process in order to expose the kernal's starch. Corn commonly contains 69 to 73 percent starch by weight. A 3.7 kilowatt (five horsepower) hammermill is located directly under a raised corn bin. A 2.38 millimeter (3/32 inch) diameter screen assures a fine, uniform grind. Refer to Table B-1 in Appendix B for typical sieve analyses of the ground corn. The screen also prevents large particles of foreign matter or whole corn kernals from plugging or damaging pumps and other system components.

Cooking

In the batch cooking process a cooker-fermenter tank is partially filled with 40 to 60^{o}C (104 to 140^{o}F) condenser cooling water from the distillation tower. A predetermined amount of ground corn is then augered into the tank. A mechnaical agitation system disperses the corn into suspension. The quantity of corn mixed with a particular quantity of tap (cooling) water is dependent upon the final glucose (and subsequently ethanol) concentration sought in the final corn-beer. For an 18 to 19 percent by weight glucose concentration (10 percent by volume ethanol concentration), a ration of three liters of tap water to each kilogram of corn (20 gallons of water to each bushel of corn) is used. This will yield 3.75 liters of corn-mash. The corn-water mixture is referred to as corn-mash prior to the beginning of fermentation.

After the corn granules are thoroughly mixed into suspension, 12 milliliters of alpha-amylase enzyme is added per 100 liters of corn-mash. The mixture should have a pH between 5.5 and 7.0. The mixture is heated by steam forced through a steel heating coil inside the tank. At about $69^{\circ}C$ ($156^{0}F$) the starch granules begin to rupture and absorb water. As the corn-mash gels, the viscosity increases substantially. The viscosity peaks at about $74^{\circ}C$ ($165^{\circ}F$) as the enzyme begins to break the long chains of starch into shorter chain dextrins (complex sugars). This solubilizes the starch and reduces viscosity. The mixture temperature is elevated to $93 \pm 3^{\circ}C$ ($199 \pm 5^{\circ}F$). This temperature is maintained for at least 30 minutes so that: one, the enzyme has sufficient time to randomly cleave (split) the starch into shorter chain dextrins and, two, the mixture is pasturized. Pasturization eliminates unwanted microbial organisms that may contaminate the fermentation process.

The mixture temperature is then lowered to $60^{\circ}C \pm 2^{\circ}C$ ($140^{\circ}F \pm 4^{\circ}F$) by running cold water through the heating coil. At this point the pH is lowered to between 3.8 and 4.2. Eight milliliters of concentrated (98 percent by weight) sulphuric acid is added to each 100 liters of corn-mash. Forty milliliters of gluco-amylase enzyme is then added for each 100 liters of corn-mash. This enzyme further breaks the short starch molecules, dextrins, into the simple sugar, glucose ($C_6H_{12}O_6$). Refer to Figure B-2 in Appendix B for a graphical presentation of the corn-mash's component change with time during cooking.

After the solution is held at $60^{\circ}C$ ($140^{\circ}F$) for four hours, it is further cooled to fermentation temperature, 28 to 32°C (82 to 90°F). The glucose-rich corn-mash is now ready for fermentation.

Fermentation

A brewer's yeast (as contrasted to Baker's yeast) called *Saccharomyces cerevsiae*, is added at the rate of 5 to 7 x 10^8 cells per liter of corn-mash. Continuous agitation is not required during the 48 hour, mostly anaerobic, fermentation process. The chemical reaction of glucose to ethyl alcohol is:

$$C_6H_{12}O_6 \rightarrow 2\ C_2H_5OH + 2\ CO_2 + 31{,}200\ \text{cal/gmole} \qquad \text{(Ogden, 1979).}$$

Due to the production of heat, cooling may be required to maintain the fermentation temperature between 28 and 32°C (82 and 90°F).

Twelve hours into the fermentation process, 40 milliliters of ammonium hydroxide (29 percent by weight solution) is added to each 100 liters of corn-beer. The base has two purposes. The first is to slightly raise the pH of the solution. The second is to provide nitrogen for the reproduction and metabolism of yeast cells. A substantial increase in fusel oil and other impurities may result if this source of nitrogen is not provided.

The corn-beer should contain ten percent (+ ½ percent) ethyl alcohol after this process and have a total solids content of about 8.5 percent by weight. Refer to Table B-2 of Appendix B for a typical proximate analysis of the final corn-beer.

THEORY AND DESCRIPTION OF DISTILLATION EQUIPMENT

Tower Function and Description

Many volumes of literature have been written which describe the distillation process in detail. The intent of this section is to review only those aspects which are necessary to interpret the results of the experimentation performed.

According to Carney (1949), distillation is a separation process utilizing differences in volatility of the components of a fluid stream. Simple distillation is the separation of components of a mixture of partial vaporization and separate recovery of the vapor and residue. Fractional distillation consists of a continuous series of vaporizations and condensations in the same column. Enriched vapor is drawn from the top while 'stripped' liquid residue is pumped from the bottom.

South Dakota State University employs a continuous flow, fractional distillation process to separate ethanol from corn-beer. The corn-beer feed is steadily pumped to the distillation tower by a positive displacement, progressive cavity pump. Refer to Appendix C for a full description of the feed pump. The tower is a two-column unit of sieve tray (perforated plate) design, 30.5 centimeters in diameter and 483 centimeters (190 inches) tall. Refer to Figures C-6 through C-10 in Appendix C for drawings and a picture of the distillation tower. This binary distillation unit operates at atmospheric pressure.

Ninety-six to 99 percent of the ethanol is boiled from the corn-beer and condensed as a 90 to 95 percent ethanol by volume aqueous solution. Figure 1 is a schematic representation of the overall function of the distillation unit.

Preheated feed is introduced at the top of the stripper column. It flows through this column at a rate of 600 to 900 liters per hour (160 to 240 gallons per hour). At the bottom of the stripper column, low pressure steam (10 N/cm^2; 15 psi) is injected. The amount of steam supplied is dictated by the temperature in the stripper column. More steam (and thus higher temperature) allows for a greater portion of ethanol to be removed from the feed. This is at the cost of a greater energy consumption.

As the steam bubles upward, it vaporizes ethanol from the corn-beer. The spent beer (stillage) is continuously removed from the bottom of the stripper where it is pumped to a centrifuge. The ethanol vapor and residual steam flow out of the stripper column and into the bottom of the rectifier column. The stripper and rectifier columns are built in a series arrangement, functioning as one unit composed of two segments.

As the ethanol and water vapor rise through the rectifier, a condensation and re-evaporation process separates the ethanol and water. Condensation is caused by the circulation of relatively cold corn-beer feed through steel coils at the top of the rectifier. The amount of cooling dictates the quantity of liquid reflux traveling down the tower. The more reflux generated, the cooler the temperature

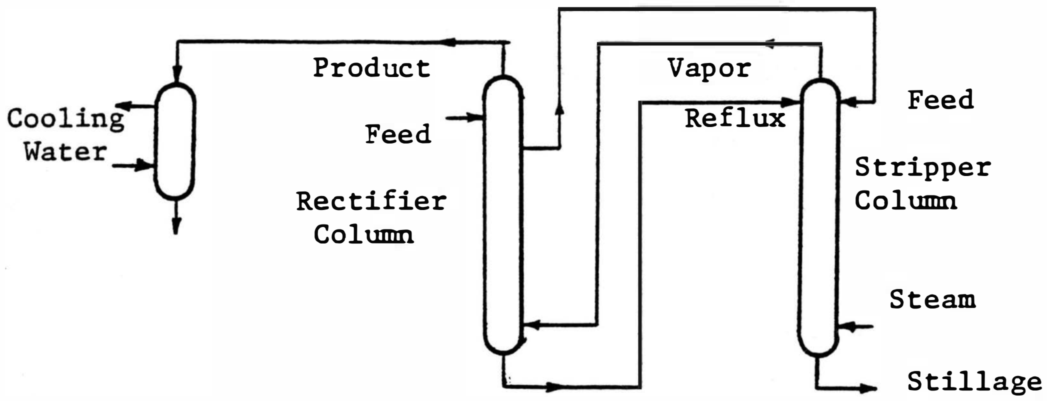


Figure 1: Typical Two-Column Fractional Distillation Unit

of the column, and the more highly concentrated the ethanol product becomes. However, once again, this is at the expense of higher steam consumption.

The preheated feed enters the stripper section at a temperature of approximately 75°C (167°F), well above that of fermentation at 30°C (86°F). Water in the rectifier condenses and drops to the bottom of the column, where it is pumped to the top of the stripper in order to remove any residual ethanol present. Refer to Appendix C for a copmlete description of the pump employed for this function. The vaporized ethanol flows off the top of the tower. It is then condensed in a water-cooled, coil condenser and removed for storage at the rate of 60 to 90 liters per hour (16 to 24 gallons per hour).

Tower Theory of Operation

The distillation unit has no moving parts. Figure 2 on the following page presents the idealized manner in which mass and heat transfer through the tower (McCabe and Smith, 1976). Vapor from the lower Tray 1 has sufficient pressure (supplied by injected steam at the bottom of the tower) to pass through perforations in Tray 1 and bubble through a layer of liquid on the top of Tray 1. Some of the vapor condenses as energy is transferred to the liquid; causing it to boil. In this way, the vapor traveling from Tray 1 to Tray 2 has higher concentration of the most volatile component, ethanol, than that which traveled into Tray 1 from below.

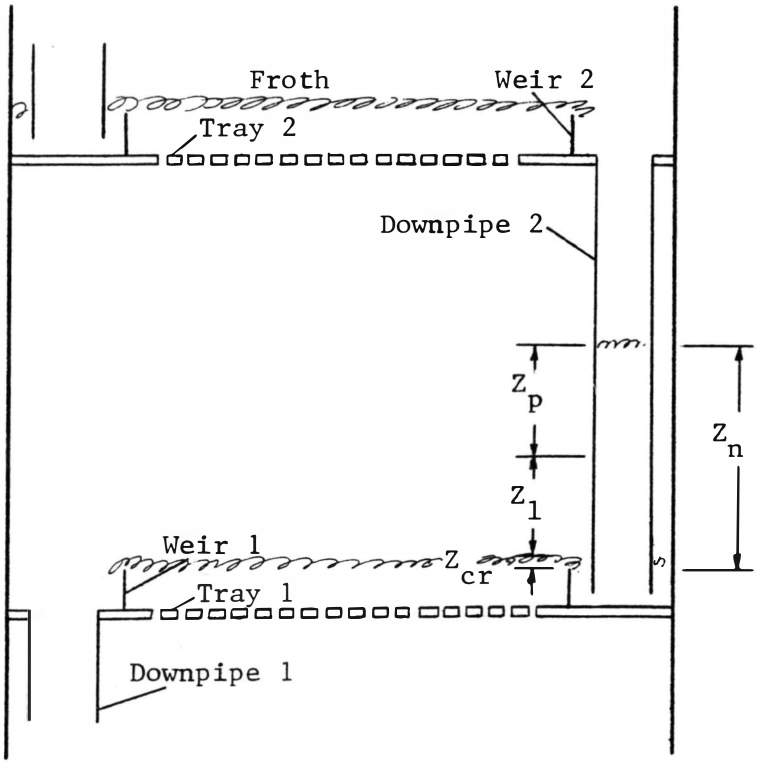


Figure 2: Internal Dynamics of a Distillation Tower

Liquid flows counter current to vapor in the process. Overflow from Weir 2 is supplied by liquid feed in the stripper column and liquid reflux in the rectifier column. Liquid flows from Tray 2 to Tray 1 by Downpipe 2. This process continues downward through the tower.

Normal operation of the sieve plate is characterized by small pressure reductions as one moves from bottom to top. Figure 2 shows the static head balance in the downpipe. Z_p provides the driving force for overcoming the pressure drop through the tray. Z_1 is the head due to friction in the flow of the liquid through and out of the downpipe. Z_{cr} is required to discharge the liquid over the weir. During operation of the tower, rates of flow must be kept low enough to keep the total head from imparing flow, that is, from exceeding the height from one weir to the next. This condition is monitored by noting any pressure increase during distillation.

This process is employed in both the stripper and rectification columns, although there are minor differences in the construction of each. For a full description of the distillation unit employed at SDSU, refer to Appendix C; Equipment.

EXPERIMENTAL PROCEDURE

The laws of conservation of mass and energy must be carefully employed in determining the operating characteristics of the distillation tower pertaining to energy consumption. Each mass and energy imput was compared with each mass and energy output of the system.

Figure 3 is a schematic representation of a distillation unit. Each fluid entering or exiting the unit was analyzed. The temperature, flow rate, and composition of the corn-beer (feed) were carefully controlled during the experiment. The rate of steam flow was the dependent variable used in the experiment. The flow rate, temperature, and composition of the stillage were monitored. The flow rate,

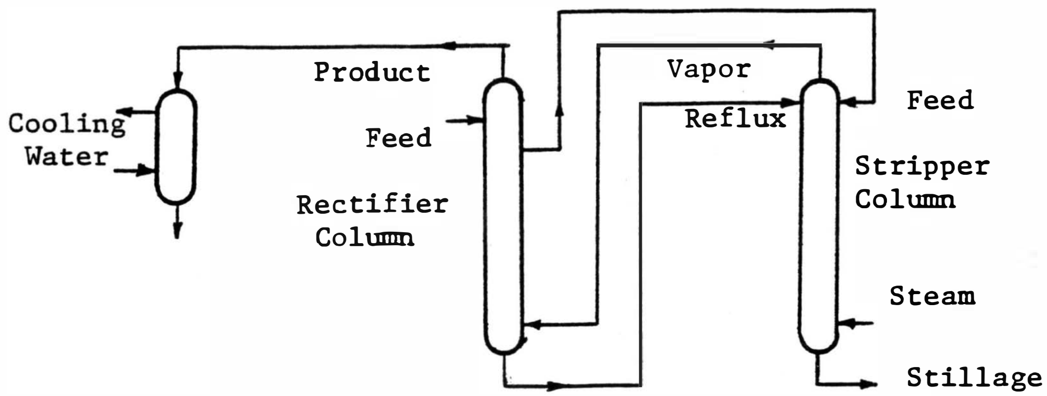


Figure 3: Typical Two-Column Fractional Distillation Unit

temperature, and percent ethanol by volume in the product were also monitored. The rate of flow of cooling water as well as its temperature rise was recorded. Much additional information was recorded throughout the system to allow for the analysis of distillation interrelationships.

There are five critical operating parameters which are most important in influencing the performance of the distillation unit. These are "corn-beer ethanol concentration," "corn-beer flow rate," "corn-beer inlet temperature," "stillage residual ethanol concentration," and "product ethanol concentration." Knowledge of how each of these parameters affect the energy requirement of distillation is of technical and economic interest.

The first operating parameter of particular interest is the "corn-beer ethanol concentration." A corn-beer with seven percent ethanol will require a different quantity of energy to distill than one containing ten percent ethanol when other factors are held constant.

The second operating parameter is the flow rate of the corn-beer into the distillation unit, "corn-beer flow rate." This determines the flow of each of the other fluid streams and will affect the overall performance of the distillation unit. Theoretically, a particular distillation unit has an optimum operational capacity for a fixed set of operating conditions and design parameters.

It is easily recognized that the temperature of the corn-beer entering the unit, "corn-beer inlet temperature," is of major consequence to the energy requirements of distillation. The preheating of corn-beer in the rectifier provides a nearly uniform inlet temperature. From knowledge of the specific heat and flow rate of the corn-beer it is a simple calculation to determine energy input when the fluid is entering the column at slightly differing temperatures. Thus, the parameter can be adequately characterized.

The temperature and flow rate of the stillage were also recorded in the experiment. The composition of the stillage, "stillage residual ethanol concentration," is a controllable parameter. With a properly designed distillation unit, the amount of residual ethanol in the stillage is dictated by the quantity of steam energy injected into the stripper column, and thus the temperature maintained in the stripper column. Economics dictate that a near optimum level of extraction be achieved. Energy is wasted if too high a degree of separation is attempted, while valuable ethanol is wasted if operation is at too low a level of separation.

The composition of the ethanol product, "product ethanol concentration," is the fifth operating parameter. The achieved proximity to the azeotropic composition of water and ethanol partially dictates the energy requirement of the separation process. This composition is controlled in distillation by varying the quantity of liquid product returned to the rectification column. Condensation of this reflux is one source of cooling in the system.

Unfortunately, time and money prohibited a careful study of all five operating parameters and the interactions which might be involved. A look at the statistical requirements of such an experiment indicates that the size is prohibitive. If three levels of treatment were made for each parameter and four replications made for error analysis, a total of 972 data points would be required. A reasonable estimate of plant operating time required to obtain a single data run is four hours, at a rough operating cost of $40 per hour. Such a project would take 324 weeks, assuming three data runs are taken a week, and cost over $150,000.

The experimental design employed is as follows. Two of the five operating parameters were independently analyzed with four replications of three treatment levels, for a total of 24 data runs. This amounted to a pair of four week experiments at a total operating cost of $3,800.

The two operating parameters chosen to be studied were "stillage residual ethanol concentration" and "corn-beer ethanol concentration" in two separate experiments. The former parameter was chosen due to its technical and economic implications. If an inadequate portion of the ethanol is extracted from the corn-beer, there is an obvious economic loss. However, too high a degree of separation wastes steam and decreases the operating capacity of the distillation tower by raising the vapor flow rate.

The following procedure was employed in experiment one to study the effects of varying concentrations of residual ethanol in the stillage upon distillation energy consumption. Ten percent ethanol in corn-beer was distilled to a 92.5 percent ethanol product at the rate of 742 liters per hour (196 gallons per hour). The corn-beer inlet temperature was maintained at 74.9^{o}C (166.8^{o}F). Since the operating temperature maintained in the stripper column is used to control "stillage residual ethanol concentration," the temperature was used to indirectly represent residual ethanol concentrations. The three levels of treatment for "stillage residual ethanol concentrations" were substituted by the operating temperature maintained in the stripper column, "center stripper temperature." The three levels employed were 94.4°C (201.9°F), 95.0 (205.0°F), and 95.6°C (204.1°F). These levels were assumed to correspond to a high, intermediate, and low level of residual ethanol in the stillage. The three temperatures were maintained by varying the quantity of steam delivered in the stripper column.

"Stillage residual ethanol concentration" was not used directly as an independent variable in experiment one. The laboratory procedure (Appendix G) to determine the "stillage residual ethanol concentration" requires two hours to perform. The results of the anlaysis were not available in time for use in operation of the distillation column. Hence, one cannot directly use results of these analyses as an operating parameter in the experiment.

The second parameter, "corn-beer ethanol concentration," was studied because of its predicted large influence upon energy consumption and because of its interrelationship with concurrent studies by the Microbiology Department. These studies involved the effects that various "corn-beer ethanol concentrations" have upon conversion efficiency (Westby and Gibbons, 1982).

The following procedure was used in experiment two to study the effect of varying concentrations of ethanol in the corn-beer, "corn-beer ethanol concentration." Corn-beer with an inlet temperature of 74.4°C (195.9°F) was refined to a 92.3 percent ethanol by volume product at the rate of 741 liters per hour (196 gallons per hour). A temperature of 95.0°C (203.0°F) was maintained in the center of the stripper column. Varying amounts of corn were added to water during the cooking process to control the ethanol concentration after fermentation. Approximately four liters of water were added to each kilogram (26 gallons of water to each bushel) of corn to achieve seven percent corn-beer. Three liters of water were added to each kilogram (20 gallons of water to each bushel) of corn to achieve ten percent corn-beer. Finally, two liters of water were added to each kilogram (14 gallons of water to each bushel) of corn to achieve 13 percent corn-beer. Small amounts of water or ethanol could then be added to the corn-beer to assure an ethanol concentration near the three levels of treatment sought.

The "corn-beer flow rate," the "corn-beer inlet temperature," and the "product ethanol concentration" will need to be studied in the future.

Data Collection

The data were collected under equilibrium conditions. The distillation tower was operated from two to five hours at the specified operating settings before data were recorded. Two criteria were established for determining adequate equilibrium conditions. First, the temperature at the center of the stripper column should deviate less than 0.15°C (0.3°F) from the specified experimental level.

Second, the temperature at the center of the rectifier should not fluctuate more than 0.3°C (0.5°F) over a period of 15 minutes. Once these conditions were met, the recording of data could begin.

Figure B-3 in Appendix B depicts the data form used for the collection of system temperatures. The temperatures during each 15 minute interval leading up to equilibrium were recorded.

Figure B-4 in Appendix B is the data form used for the remainder of data collection. Data were recorded at 30 minute intervals leading up to equilibrium conditions. The steam rate of flow was measured by the orifice and differential pressure cell. Flow rates of corn-beer feed, reflux, stillage, cooling water, and ethanol product were determined by volume per unit time (with a stop watch and bucket). The amount of ethanol in the product was measured by a hydrometer. The steam pressure was measured by a bourdon tube type gauge and was used in determining the rate of energy flow. Atmospheric pressure was an uncontrolled variable studied for possible effects in the distillation process. Data was acquired from the SDSU Agricultural Engineering Department which works in conjunction with the National Oceanic and Atmospheric Administration in gathering weather data. The data were taken from a USWB No. 553-2½ barograph provided by Freiz Baltimore. Electrical measurements were not used in the experimentation. "Stillage residual ethanol concentration" was determined by a laboratory analysis described in Appendix G. The sample taken for this analysis was acquired after equilibrium conditions were achieved. The last recordings of all the other measurements were considered instantaneous, steady state readings.

Analysis Procedure

The effects that the two chosen operating parameters have upon the magnitude of consumed energy were studied in a statistical manner. The two experiments performed were designed independently such that interactions between the independent variables were not studied.

Four replications of three levels of treatment were made for each of the operating parameters. A statistical multiple regression was independently performed involving each parameter and the energy requirement for the three levels of treatment. With "energy per liter ethanol" as the dependent variable, a total of seven independent variables, suspected of being related to energy consumption, were analyzed in the models. The sensitivity of the statistical test dictated that only seven variables be employed in the multiple regression analyses because of the limited number of observations (12) in each of the experiments. These seven are presented in Table 3 for each experiment.

Table 3. Selected Variables Employed in the Multiple Regression Analyses

EXPERIMENT ONE	EXPERIMENT TWO
Dependent Variable:	Dependent Variable:
energy per liter ethanol	energy per liter ethanol
Independent Variables:	Independent Variables:
center stripper temperature	corn-beer ethanol concentration
corn-beer ethanol concentration	top rectifier temperature
stillage residual ethanol concentration	stillage residual ethanol concentration
product flow rate	atmospheric pressure
product ethanol concentration	product ethanol concentration
center rectifier temperature	center rectifier temperature
stillage flow rate	stillage flow rate

A number of statistical terms should be defined for interpretation of the results. The statistic F is employed in determining if a particular independent variable is statistically significant in the prediction equation. In decimal form PROB F is the probability that the conclusion of significance is in error. A PROB F of less than 0.05 but greater than 0.01 indicates the relationship under study is statistically significant. A PROB F of less than 0.01 indicates that the probability the conclusion reached is in error is less than one percent, and it is said that the statistic is highly significant. According to Walpole and Myers (1978), an estimate of the square of the correlation coefficient, ρ^2, is given by the square of the sample correlation coefficient, R^2. Always remaining positive and less than one, R^2 is the decimal form of the percent of variation in the dependent variable that is explained by the model under consideration.

To investigate interrelationships in the operation of the distillation tower, statistical correlation was determined among each of 22 system variables. The variables are listed and defined in Table B-3 in Appendix B.

Data were also collected for the purpose of partitioning the expended energy into meaningful components. These components include the energy required to heat the corn-beer to an adequate level for boiling off the ethanol ("corn-beer heating"), the energy involved with providing reflux within the rectifier column ("reflux generation"), the energy involved with the condensation of the ethanol product ("product condensation"), and the thermal energy losses incurred by the system ("system thermal losses"). The latter is found by the difference between total energy of distillation and the other three components. Hence, it includes all errors in the analysis.

RESULTS AND DISCUSSION

This section presents results obtained from statistical analyses of the experimental data. The data are first presented and the dependent variable defined. Next, results of linear and multiple regression analyses are employed to show how the dependent variable, "energy per liter ethanol," is a function of "center stripper temperature" under the conditions of the first experiment. Then, results of linear and multiple regression analyses are employed to show how "energy per liter ethanol" is a function of "corn-beer ethanol concentration" under the conditions of the second experiment. Correlations are presented which show interrelationships of various measured variables in the system. Finally, steam consumption, as measured by "energy per liter ethanol," is partitioned into four components.

Data

Numerical values of 22 variables have been assembled for use in the statistical analyses. The data are presented in Table B-4 in Appendix B. The dependent variable in each of these experiments is listed in the eighth column. "Energy per liter ethanol" is the quotient of "energy flow rate" and "product anhydrous ethanol equivalent flow rate." Therefore, the response in the experiments is based on an anhydrous-equivalent liter of ethanol.

Regression Analyses For Experiment One

A multiple regression analysis was performed between the dependent variable, "energy per liter ethanol," and the seven independent variables of experiment one. The parameter, "center stripper temperature," was the best single-variable model found by the computer analysis. Table 4 is a summary of the correlations between the dependent variable and each of the seven independent variables studied in the multiple regression analysis.

Table 4: Correlations Between "Energy Per Liter Ethanol" and Seven Independent Variables in Experiment One

VARIABLE	R^2	PROB F
Center Stripper Temperature	0.53	0.007**
Corn-beer Ethanol Concentration	0.01	0.742
Stillage Residual Ethanol Concentration	0.07	0.389
Product Flow Rate	0.01	0.713
Product Ethanol Concentration	0.06	0.458
Center Rectifier Temperature	0.08	0.370
Stillage Flow Rate	0.08	0.368

**Statistically highly significant, one percent level.

Significant findings of the multiple regression analysis are presented in Table 5. The square of the estimate of the correlation coefficient, R^2, is 0.53; indicating that 53 percent of the variation in "energy per liter ethanol" is explained by changes in "center stripper temperature." Figure 4 presents a graphical presentation of the data and this single-variable relationship.

Both bivariate and trivariate regression models showed

Table 5: Best Regression Models for Experiment One, "Center Stripper Temperatures" Serving as the Parameter Under Study

BEST SINGLE-VARIABLE REGRESSION MODEL: $R^2 = 0.53$

	PARAMETER ESTIMATE	F
Intercept:	-14.453	
Center Stripper Temperature, X:	0.192	37.667**

$$Y = -14.453 + 0.192(X)$$

BEST BIVARIATE REGRESSION MODEL: $R^2 = 0.72$

	PARAMETER ESTIMATE	F
Intercept:	-18.868	
Center Stripper Temperature, X:	0.213	37.667**
Center Rectifier Temperature, Z:	0.0271	12.807*

$$Y = -18.868 + 0.213(X) + 0.0271(Z)$$

BEST TRIVARIATE REGRESSION MODEL: $R^2 = 0.88$

	PARAMETER ESTIMATE	F
Intercept:	-19.915	
Center Stripper Temperature, X:	0.246	37.667**
Center Rectifier Temperature, Z:	0.0310	12.807*
Product Flow Rate, W:	-0.0316	11.328*

$$^{\#}Y = -19.915 + 0.246(X) + 0.0310(Z) - 0.0316(W)$$

#Recommended Model.

statistical improvement over the single-variable model. The best bivariate model uses "center stripper temperature" and "center rectifier temperature" as a prediction of energy consumption with an R^2 of 0.72.

The trivariate model employs "center stripper temperature," "center rectifier temperature" and "product flow rate" to yield an R^2 of 0.88.

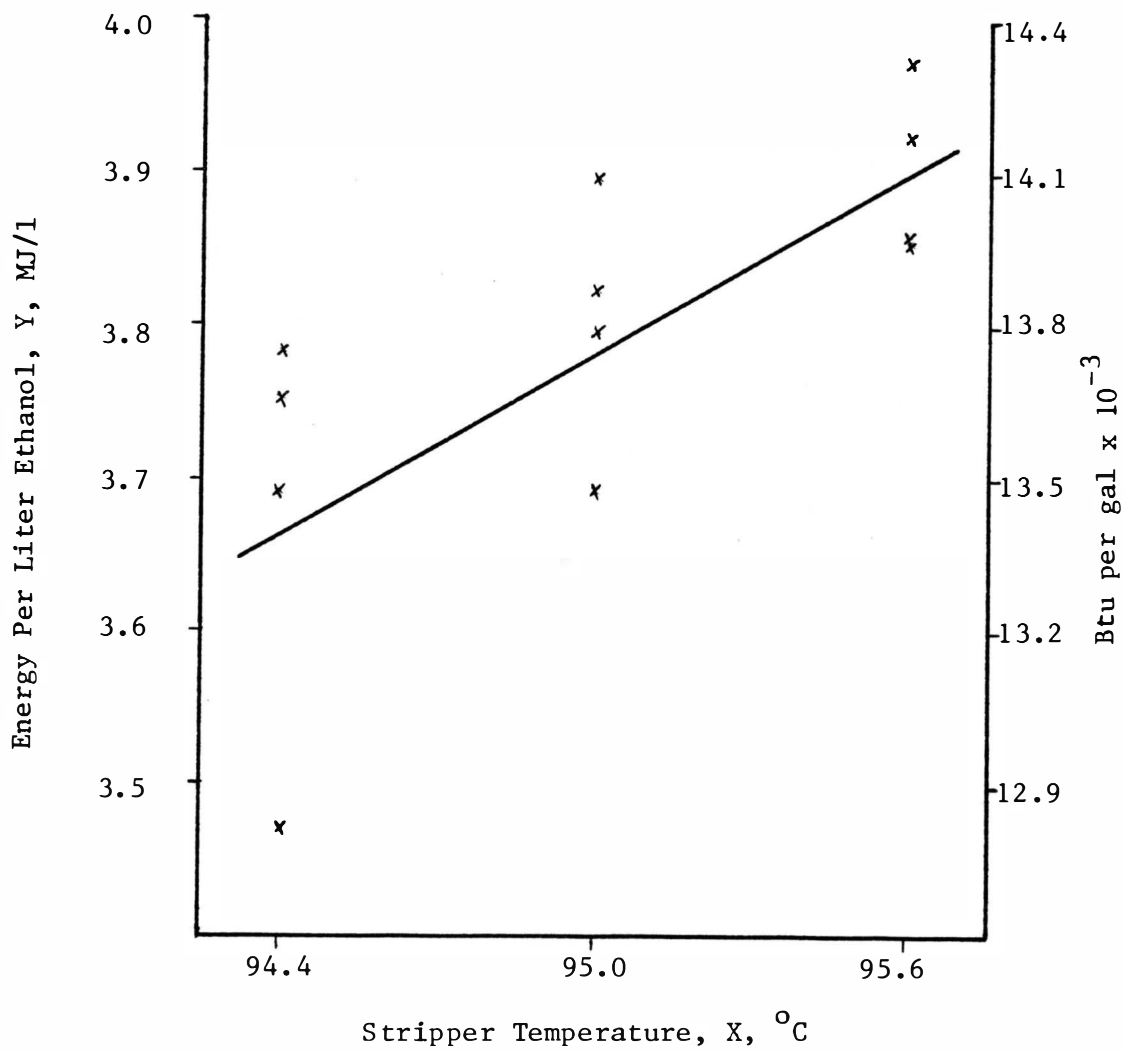


Figure 4: Best Single-Variable Linear Regression Model for Experiment One

Uncontrolled variations in "center rectifier temperature" and "product flow rate" significantly influence the dependent variable. It is recommended that all three independent variables be employed for the prediction of "energy per liter ethanol" under conditions similar to those of experiment one. An effort to use "center stripper temperature" as a quadratic model to predict "energy per liter ethanol" showed that model was statistically insignificant.

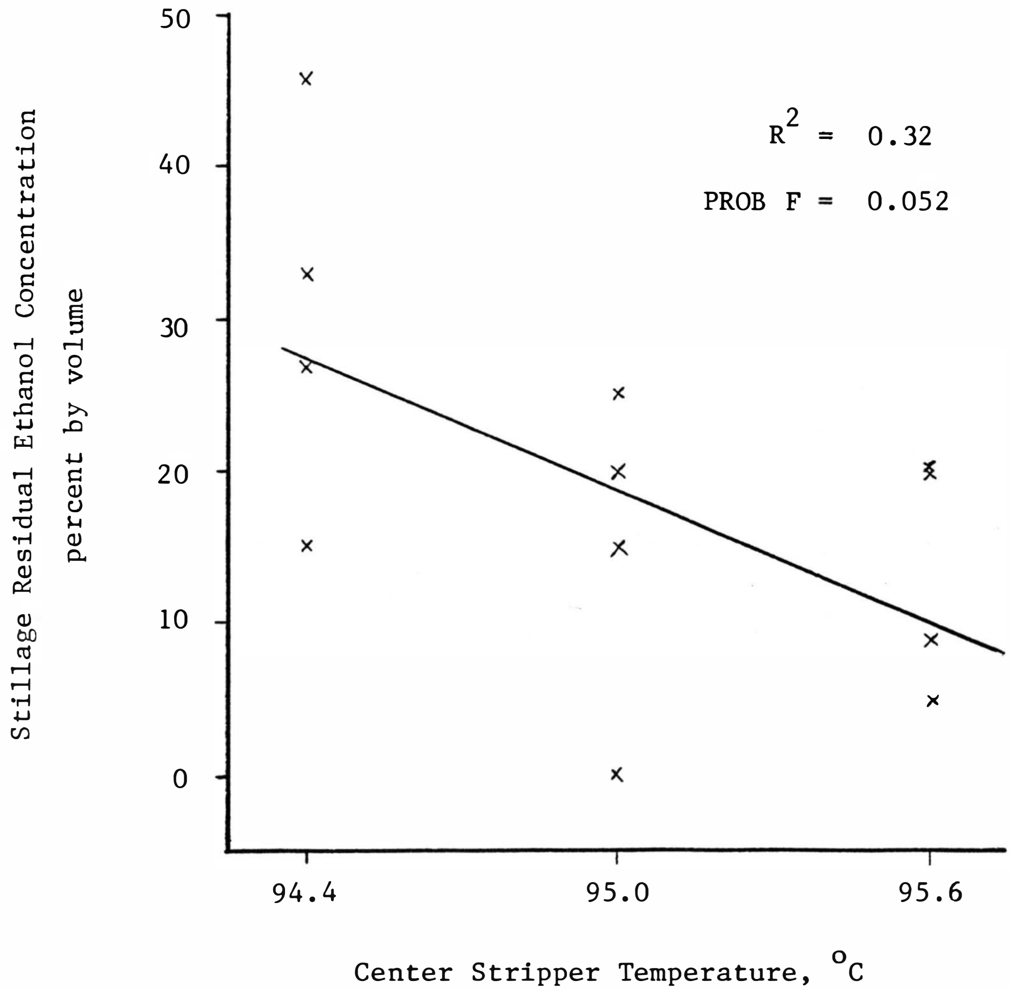


Figure 5: Evidence of the Inverse Relationship Between "Stillage Residual Ethanol Concentration" and "Center Stripper Temperature" in Experiment One

The assumed inverse relationship between "stillage residual ethanol concentration" and "center stripper temperature" settings was not well established. As indicated in Figure 5, only 32 percent of the variation in "stillage residual ethanol concentration" was explained by "center stripper temperature." A likely source of the problem is the large random error associated with the laboratory analysis method for determining "stillage residual ethanol concentration" (see Appendix G).

A more accurate method is described by Gunness and Baker (1938) and involves the controlled oxidation of ethanol to acetic acid by dichromate in an acid solution. This method is said to give acceptable results at dilutions down to 0.01 percent of ethanol by weight in the stillage.

Regression Analyses for Experiment Two

A multiple regression analysis between "energy per liter ethanol" and the seven independent variables of experiment two produced the following results. Table 6 indicates the single-variable relationships between the dependent variable and each of the seven independent variables. The experimental parameter, "corn-beer ethanol concentration," produced a statistically highly significant single-variable model. Table 7 presents multiple regression results. In

Table 6: Correlation Between "Energy per Liter Ethanol" and Seven Independent Variables in Experiment Two

VARIABLE	R^2	PROB F
Corn-Beer Ethanol Concentration	0.94	0.001**
Top Rectifier Temperature	0.02	0.658
Atmospheric Pressure	0.04	0.526
Stillage Residual Ethanol Concentration	0.17	0.176
Product Ethanol Concentration	0.42	0.023
Center Rectifier Temperature	0.12	0.280
Stillage Flow Rate	0.01	0.997

**Statistically highly significant, one percent level

the case of the simple model in experiment two, R^2 was 0.94, indicating that 94 percent of the variations in "energy per liter ethanol" is explained by changes in "corn-beer ethanol concentration."

Results of higher order analysis indicate that only the addition of the square of "corn-beer ethanol concentration" is statistically significant. In this case, R^2 is 0.99. Figure 6 is a graphic representation of the quadratic model. This model is recommended for the prediction of "energy per liter ethanol" under conditions similar to those of experiment two.

Table 7: Best Regression Models for Experiment Two, "Corn-Beer Ethanol Concentration" Serving as the Parameter Under Study

BEST SINGLE-VARIABLE REGRESSION MODEL:	$R^2 = 0.94$	
	PARAMETER ESTIMATE	F
Intercept:	6.836	
Corn-Beer Ethanol Concentration, X:	-0.286	619.00**
$Y = 6.836 - 0.286(X)$		
BEST QUADRATIC REGRESSION MODEL:	$R^2 = 0.99$	
	PARAMETER ESTIMATE	F
Intercept:	9.923	
Corn-Beer Ethanol Concentration, X:	-0.940	619.00**
Square of Corn-Beer Ethanol Concentration, (X^2):	0.0324	28.07**
#$Y = 9.923 - 0.940(x) + 0.0324(X^2)$		

#Recommended Model

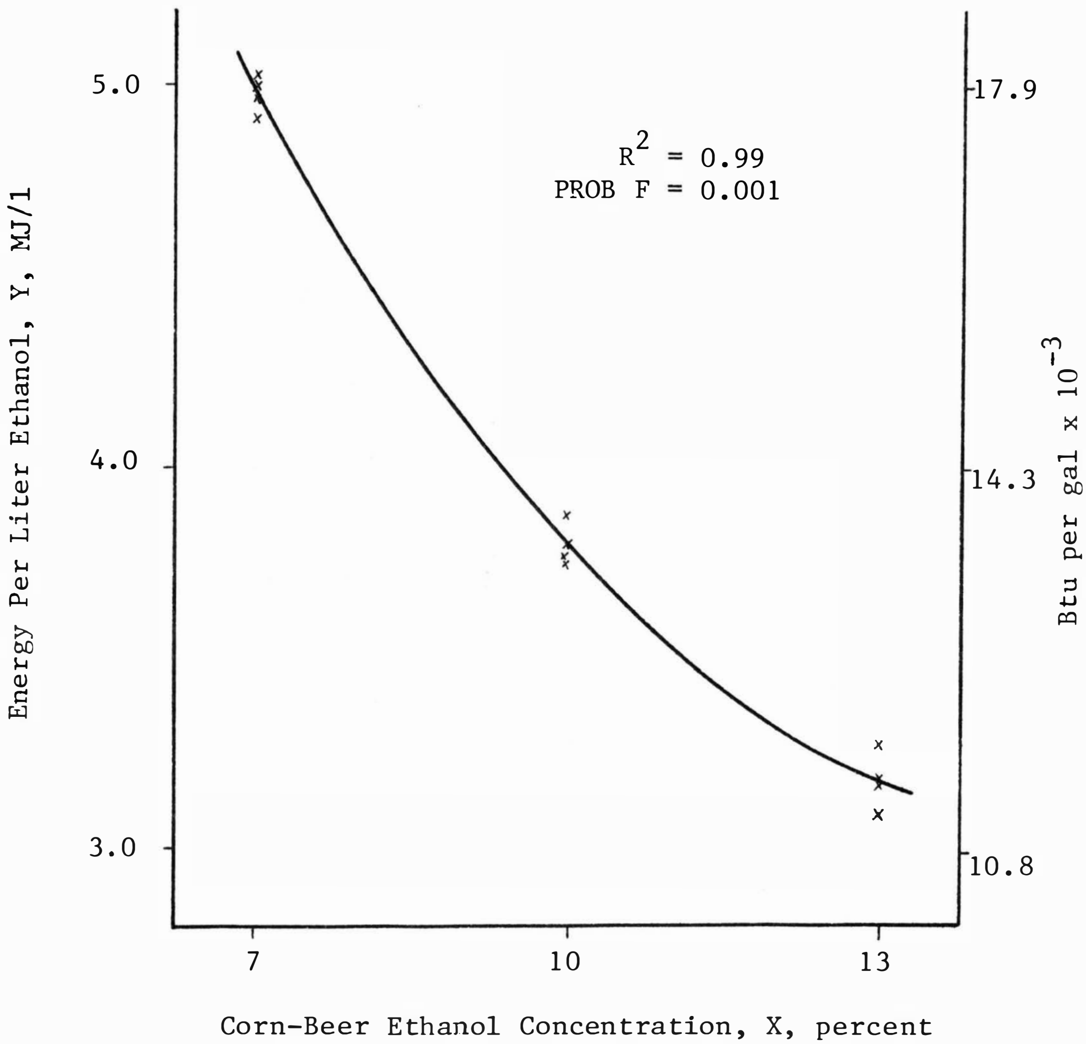


Figure 6: Quadratic (Best) Regression Model for Experiment Two

Correlation

Sample correlation coefficients have been generated among the 22 variables of each experiment. The objective of this effort is to study interrelationships in the distillation process and so to better understand that process. Tables B-5 and B-6 present the correlation coefficients and the PROB F associated with each. Tables 9 and 10 present the statistically significant results of this analysis. With each correlation is given one possible reason for the relationship. However, one must not construe correlation as a cause and effect relationship as there is no evidence for such a relationship.

One of the most intriguing relationships found was that between "atmospheric pressure" and "product ethanol concentration." Since the boiling point of ethanol-water solutions are functions of overhead pressure, the rectifier temperatures correlate with atmospheric pressure. Operation of the tower was then adjusted to compensate for these variations in temperature, resulting in varying product ethanol concentrations.

The values in Table 8 indicate that between 0.2 and 10.9 percent of the ethanol in the corn-beer is not recovered during distillation. This loss is the difference between the laboratory analysis of the corn-beer (performed on samples taken during distillation) and the flow of anhydrous-equivalent ethanol from the distillation unit. Only one to three percentage points is due to ethanol lost in the spent stillage.

The correlation results of Table B-5 in Appendix B for experiment one indicate that only the "product anhydrous ethanol equivalent

Table 8: Apparent Ethanol Loss During Distillation

	apparent ethanol loss, liters per hour	apparent ethanol loss, percent of total flow
EXPERIMENT 1		
1	7.50	10.87
2	0.13	0.17
3	2.97	4.21
4	4.38	6.06
5	6.66	9.24
6	6.95	9.85
7	2.60	3.50
8	1.35	1.86
9	2.47	3.51
10	2.55	3.50
11	4.50	6.23
12	2.63	3.49
average	3.73	5.21
EXPERIMENT 2		
1	1.70	3.48
2	6.43	9.34
3	1.55	2.20
4	8.83	9.43
5	2.22	3.18
6	2.55	2.77
7	5.46	5.80
8	3.80	5.22
9	4.34	9.13
10	8.87	10.25
11	3.49	7.59
12	2.28	4.76
average	4.29	6.10

Table 9: Statistically Significant Correlations for Experiment One; "Center Stripper Temperature" Serving as the Ewperimental Variable.

STATISTICALLY SIGNIFICANT CORRELATION EXISTS BETWEEN THESE QUANTITIES:	'r'	PROB F	REASON FOR CORRELATION
corn-beer ethanol concentration reflux flow rate	0.590	0.0434*	unknown
stillage residual ethanol concentration center rectifier temperature	0.616	0.0329*	the "product ethanol concentration" sought is determined by the reflux generated in the rectifier
stillage residual ethanol concentration stillage flow rate	-0.751	0.0048**	column may be more efficient at higher flow rates
stillage residual ethanol concentration reflux temperature	0.786	0.0024**	unknown
energy flow rate energy per liter ethanol	0.805	0.0016**	column operation is at near constant rates of flow and "corn-beer ethanol concentrations"
energy flow rate center stripper temperature	0.842	0.0006**	increasing the "center stripper temperature" to extract more ethanol requires more energy
product flow rate product anhydrous ethanol equivalent flow rate	0.961	0.0001**	same stream
product ethanol concentration top rectifier temperature	-0.583	0.0465*	"product ethanol concentration" is a function of the boiling point of the water-ethanol mixture
product ethanol concentration center stripper temperature	0.583	0.0454*	column may be more efficient at higher vapor flow rates
energy per liter ethanol center stripper temperature	0.730	0.0070**	increasing "center stripper temperature" to extract more ethanol requires more "energy per liter ethanol"
top rectifier temperature corn-beer inlet temperature	-0.592	0.0426*	unknown
stillage flow rate reflux temperature	-0.771	0.0034**	unknown
corn-beer flow rate corn-beer inlet temperature	-0.613	0.0340*	unknown
corn-beer inlet temperature water inlet temperature	0.632	0.0276*	as winter approaches each get colder
relux flow rate center stripper temperature	0.712	0.0093**	varying the "center stripper temperature" requires differing amounts of reflux to maintain product quality
apparent ethanol loss product anhydrous ethanol equivalent flow rate	-0.616	0.0330*	unknown
water flow rate water outlet temperature	-0.900	0.0001**	heat balance

* statistically significant, 5 percent level
** statistically highly significant, 1 percent level

Table 10: Statistically Significant Correlations for Experiment Two; "Corn-Beer Ethanol Concentration" Serving as the Parameter Under Study

STATISTICALLY SIGNIFICANT CORRELATION EXISTS BETWEEN THESE QUANTITIES:	'r'	PROB F	REASON FOR CORRELATION
atmospheric pressure product ethanol concentration	0.666	0.0182*	the water-ethanol boiling point in the rectifier dictates the "product ethanol concentration"
atmospheric pressure center rectifier temperature	-0.614	0.0338*	"atmospheric pressure" dictates the water-ethanol boiling point; the inverse relationship is not understood
atmospheric pressure top rectifier temperature	0.717	0.0086**	"atmospheric pressure" dictates the water-ethanol boiling point
atmospheric pressure corn-beer outlet temperature	0.621	0.0312*	the rectifier temperature dictates the temperature of the corn-beer leaving the rectifier preheater
corn-beer ethanol concentration energy flow rate	0.978	0.0001**	a higher "corn-beer ethanol concentration" requires a higher steam energy flow rate to the tower
corn-beer ethanol concentration product flow rate	0.991	0.0001**	when more ethanol is introduced to the tower, more product is realized
corn-beer ethanol concentration product ethanol concentration	-0.701	0.0112*	the tower has less water to separate from the ethanol
corn-beer ethanol concentration product anhydrous ethanol equivalent flow rate	-0.991	0.0001**	when more ethanol is introduced to the tower more ethanol in the product is realized
corn-beer ethanol concentration energy per liter ethanol	-0.971	0.0001**	less energy is required when a tower has less water to separate from the ethanol
corn-beer ethanol concentration corn-beer inlet temperature	-0.917	0.0001**	"corn-beer inlet temperature" was purposely varied to control the quantity of reflux generated
corn-beer ethanol concentration corn-beer outlet temperature	-0.597	0.0405*	"corn-beer ethanol concentration" affects rectifier capacity and therefore temperature
corn-beer ethanol concentration water outlet temperature	0.703	0.0108*	with higher "corn-beer ethanol concentrations" more total steam energy and so cooling is needed
corn-beer ethanol concentration apparent ethanol loss	0.627	0.0292*	unknown
stillage residual ethanol concentration center rectifier temperature	-0.726	0.0075**	as the "center rectifier temperature" decreases, more ethanol is forced through the stripper
energy flow rate product flow rate	0.992	0.0001**	more ethanol moving through the column requires more total steam energy
energy flow rate product ethanol concentration	-0.729	0.0071**	more energy is required for greater product refinement
energy flow rate product anhydrous ethanol equivalent flow rate	0.991	0.0001**	more ethanol moving through the column requires more total steam energy
energy flow rate energy per liter ethanol	-0.981	0.0001**	"corn-beer ethanol concentration" dictates total steam, but less water is separated
energy flow rate corn-beer inlet temperature	-0.907	0.0001**	"corn-beer inlet temperature" was purposely varied according to the "corn-beer ethanol concentration"

Table 10: Continued

STATISTICALLY SIGNIFICANT CORRELATION EXISTS BETWEEN THESE QUANTITIES:	'r'	PROB F	REASON FOR CORRELATION
energy flow rate conr-beer outlet temperature	-0.620	0.0314*	"corn-beer outlet temperature" depends upon the rectifier temperature, and this corresponds to the "corn-beer ethanol concentration," which dictates the "energy flow rate"
energy flow rate stillage temperature	0.601	0.0386*	more energy introduced to the tower yielded a higher "stillage temperature"
energy flow rate water outlet temperature	0.759	0.0042**	more steam energy into the system dictates more energy must flow out of the system
corn-beer inlet temperature apparent ethanol loss	-0.639	0.0254*	unknown
corn-beer outlet temperature water flow rate	-0.592	0.0424*	unknown
stillage temperature water inlet temperature	-0.580	0.0482*	unknown
stillage temperature water inlet temperature	0.942	0.0001**	atmospheric pressure could influence both of these
stillage temperature water outlet temperature	0.886	0.0001**	each are functions of the total steam energy flow rate
water flow rate water inlet temperature	-0.690	0.0149*	unknown
water flow rate water outlet temperature	-0.588	0.0443*	the higher the flow rate the lower the temperature rise needed to remove the same energy
water inlet temperature water outlet temperature	0.794	0.0020**	the higher the inlet temperature, the higher the outlet temperature for equal energy removal
product ethanol concentration center rectifier temperature	-0.815	0.0012**	the water-ethanol boiling point in the rectifier dictates the "product ethanol concentration"
product ethanol concentration corn-beer inlet temperature	0.603	0.0375*	"corn-beer inlet temperature" was purposely varied according to "corn-beer ethanol concentration"; "product ethanol concentration" correlates with this
product anhydrous ethanol equivalent flow rate energy per liter ethanol	-0.986	0.0001**	the ethanol flow rate is dictated by "corn-beer ethanol concentration"; which determines energy consumption
product anhydrous ethanol equivalent flow rate corn-beer inlet temperature	-0.894	0.0001*	"corn-beer inlet temperature" was purposely varied according to "corn-beer ethanol concentration"; the ethanol flow rate correlates with this
product anhydrous ethanol equivalent corn-beer outlet temperature	-0.578	0.0493*	"corn-beer outlet temperature correlates with "corn-beer ethanol concentration"; which dictates ethanol flow rate
product anhydrous ethanol equivalent flow rate water outlet temperature	0.759	0.0042**	ethanol flow rate is dictated by "corn-beer ethanol concentration"; which correlates with the rectifier temperature and to "water outlet temperature"
energy per liter ethanol corn-beer inlet temperature	0.880	0.0002**	"corn-beer inlet temperature was purposely varied according to "corn-beer ethanol concentration"; "energy per liter ethanol" correlates with this

Table 10: continued

STATISTICALLY SIGNIFICANT CORRELATION EXISTS BETWEEN THESE QUANTITIES:	'r'	PROB F	REASON FOR CORRELATION
energy per liter ethanol stillage temperature	-0.624	0.0303*	less energy consumed dictates a lower "stillage temperature"
energy per liter ethanol water outlet temperature	-0.826	0.0009**	"energy per liter ethanol" correlates with "energy flow rate" which dictates the energy to be removed
center rectifier temperature stillage temperature	0.581	0.0477**	atmospheric pressure could influence both of these
stillage flow rate water flow rate	-0.623	0.0305*	unknown
corn-beer inlet temperature corn-beer outlet temperature	0.704	0.0107*	the higher the inlet temperature, the outlet temperature for equal energy removal
product flow rate product ethanol concentration	-0.699	0.0114*	steam energy correlates with "corn-beer ethanol concentration," so there is less water to be separated
product flow rate product anhydrous ethanol equivalent flow rate	-0.999	0.0001**	these are the same fluid streams
product flow rate energy per liter ethanol	-0.987	0.0001**	"product flow rate" correlates with "corn-beer ethanol concentration," so there is less water to be separated
product flow rate corn-beer inlet temperature	-0.899	0.0001**	"corn-beer inlet temperature" is based on "corn-beer ethanol concentration"; which dictates product flow
product flow rate corn-beer outlet temperature	-0.583	0.0478*	"product flow rate" depends on "corn-beer ethanol concentration"; which dictates the rectifier temperature and so "corn-beer outlet temperature"
product flow rate stillage temperature	0.577	0.0496*	"product flow rate" correlates with "energy flow rate"; which dicates "stillage temperature"
product flow rate water outlet temperature	-0.760	0.0041**	"product flow rate" correlates with flow rate"; which dictates more energy to be removed
product ethanol concentration product anhydrous ethanol equivalent flow rate	-0.692	0.0126*	lower ethanol flow rates allow for better separation in the rectifier
product ethanol concentration energy per liter ethanol	0.646	0.0232*	a higher degree of separation requires more energy

* Statistically significant, five percent level
** Statistically highly significant, one percent level

flow rate" correlates with this loss. From Table B-6 in Appendix B, for experiment two the correlation results suggest a relationship between the "apparent ethanol loss" and "corn-beer ethanol concentration" as well as "corn-beer inlet temperature."

It is not apparent as to where the discrepancy occurs. Measurements of ethanol concentrations or flow rates of fluid streams are likely sources of error. There is no correlation between "water outlet temperature" and "apparent ethanol loss"; ruling out this likely possibility.

Components of Steam Consumption

Steam consumption, as measured by "energy per liter ethanol," has been partitioned into four components. These are "corn-beer heating," "reflux generation," "product condensation," and "system thermal losses." Results from this analysis are presented in Table 11. "Corn-beer heating" required, on average, 20 percent of the distillation steam consumed; "reflux generation," 38 percent; "product condensation," 18 percent; and "system thermal losses," 24 percent.

Of particular interest in Table 11 is the fact that, for experiment two, "system thermal losses" per liter of ethanol increases in the case when seven percent corn-beer is used (rows 1, 9, 11, 12) and decreases in the case when 13 percent corn-beer is used (rows 4, 6, 7, 10). As anticipated, the energy given up for the condensation of the product, "product condensation," remained constant when measured on the basis of a liter of ethanol.

Table 11: Partitioned "Energy Per Liter Ethanol" in Megajoules Per Liter

	corn-beer heating	reflux generation	product condensation	system thermal losses
		MJ/l		
EXPERIMENT 1				
1	0.79	1.43	0.80	0.88
2	0.69	1.21	0.76	0.81
3	0.73	1.40	0.66	1.05
4	0.66	1.37	0.70	0.96
5	0.66	2.20	0.05	0.95
6	0.79	1.28	1.04	0.64
7	0.76	1.58	0.43	1.20
8	0.82	1.50	0.66	0.87
9	0.83	1.39	0.74	0.84
10	0.78	1.43	0.74	0.84
11	0.79	1.32	0.70	0.89
12	0.77	1.57	0.71	0.88
average	0.76	1.47	0.67	0.90
percent of total	20	39	17	24
EXPERIMENT 2				
1	1.18	1.90	0.60	1.20
2	0.86	1.43	0.74	0.85
3	0.85	1.37	0.72	0.83
4	0.65	1.05	0.78	0.63
5	0.85	1.38	0.71	0.80
6	0.66	1.07	0.74	0.65
7	0.66	1.09	0.77	0.60
8	0.83	1.35	0.76	0.81
9	1.03	1.96	0.72	1.29
10	0.56	1.17	0.73	0.81
11	0.94	1.93	0.69	1.47
12	0.95	1.92	0.67	1.44
average	0.84	1.47	0.73	0.95
percent of total	21	37	18	24

The possibilities for reducing each of these four components of steam consumption are as follows. By use of hot stillage in a heat exchanger between the rectifier and the stripper columns, the "corn-beer heating" requirement might be cut in half. "Reflux generation" can be reduced under two conditions. First, improvements in the design or construction of the rectifier column will decrease the amount of reflux required for a given separation. Also, if a lower "product ethanol concentration" can be tolerated, then less reflux and thus less energy will be required. "Product condensation" is one area where the energy requirement cannot be reduced. However, if the energy from the condenser water can be reused, as in the cooking process, then increased plant efficiency can be anticipated. The stripper and rectifier columns are insulated with fiberglass. However, a reduction in "system thermal losses" may be realized with further insulation of the distillation apparatus.

The reflux ratio of the distillation process is defined as the ratio of the quantity of liquid ethanol returned to the top of the rectifier column to the quantity of ethanol product removed from the process. It is a measure of the efficiency of the distillation process and can be determined by dividing the energy of "reflux generation" by "product condensation." However, since significant "system thermal losses" are involved, the reflux ratio has not been estimated for the distillation tower at SDSU. High "system thermal losses" induce error in the calculations of reflux ratio, invalidating the values obtained. These losses are believed to be responsible for the irregularities of the data for row five in experiment one as shown in Table 11.

Results of correlation analyses between the dependent variable "energy per liter ethanol" and each of these four components in experiment one yield the results in Table 12. None of the four components independently explained a statistically significant portion of the steam requirement for the first experiment. Hence, knowledge of a component of "energy per liter ethanol" cannot be employed to predict the total steam requirement on the basis of this study.

A totally different picture emerges in the second experiment. Table 13 indicates that the component, "reflux generation," predicts 99 percent of the variation in "energy per liter ethanol." Hence, by measuring this single component, one may be able to predict the total steam requirement under similar operating conditions.

The reason for the difference in the significance of the results in Tables 12 and 13 can be observed from Figures 4 and 6. In experiment one, the steam consumption varied (from 3.5 to 4.0 megajoules per liter) much less than in experiment two (from 3.0 to 5.0 megajoules per liter). Hence, the sensitivity of the analysis of experiment one is much poorer than that of experiment two.

Correlation data were generated among the four components of energy consumption as well as between them and the 22 variables studied earlier. The analysis was performed, but did not yield any helpful relationships. The correlation coefficients are listed in Tables B-7 and B-8, Appendix B.

The results compiled herein are based upon a limited number of observations. Further study is required before a complete understanding

Table 12: Correlations Between "Energy Per Liter Ethanol" and the Four Components of Partitioning of Experiment One

COMPONENT	R^2	PROB F
Corn-Beer Heating	0.11	0.281
Reflux Generation	0.28	0.079
Product Condensation	0.12	0.279
System Thermal Losses	0.21	0.135

Table 13: Correlations Between "Energy Per Liter Ethanol" and the Four Components of Partitioning of Experiment Two

COMPONENT	R^2	PROB F
Corn-Beer Heating	0.80	0.001**
#Reflux Generation, X	0.99	0.001**
Product Condensation	0.62	0.002**
System Thermal Losses	0.93	0.001**

#Yields regression equation of $Y = 2.198 + 0.750(X)$

**Statistically highly significant, one percent level.

of distillation interrelationships is obtained. Since only five months of intermittent operation has been realized, further experimentation is recommended before long-term conclusions are drawn about the performance of the machinery under investigation.

SUMMARY AND CONCLUSIONS

Steam energy consumption of small-scale fractional distillation at the South Dakota State University Alcohol Fuels Research Facility was determined. The two-column unit under study is of sieve tray design and operates at atmospheric pressure. The stainless steel tower is 30.5 centimeters (12 inches) in diameter and 483 centimeters (190 inches) tall. It is capable of refining 90 liters (24 gallons) per hour of 92.5 percent ethanol product from a ten percent ethanol corn-beer.

Five operating parameters considered to be most important in influencing the performance of the distillation tower are the composition of the corn-beer feed, the flow rate of the corn-beer feed, the inlet temperature of the corn-beer feed, the composition of the spent stillage, and the composition of the ethanol product. Two of these, the composition of the spent stillage and the composition of the corn-beer feed, were studied in experiments one and two, respectively, to determine effects upon the energy consumption of the distillation process. A total of 22 system variables were studied in each experiment.

In the first experiment, the energy requirement for distillation was studied by varing the concentration of ethanol in the spent stillage. This was done by setting the temperature at the center of the stripper column at 94.4°C (201.9°F), 95.0°C (203.0°F), and 95.6^{o}C (204.1^{o}F). With the conditions of experiment one, an inverse relationship (R^2 = 0.88) was developed between the energy requirement of distillation and the temperature at the center of the stripper column when the variables "center rectifier temperature" and "product ethanol concentration" were included in the model. An effort to establish the assumed relationship between "stillage residual ethanol concentration" and "center stripper temeprature" proved inconclusive, however.

In the second experiment, the energy requirement for distillation was studied by setting the concentration of ethanol in the corn-beer feed at 7, 10, and 13 percent by volume. With the conditions of experiment two, an inverse, quadratic relationship (R^2 = 0.99) was developed between the energy requirement of distillation and the "corn-beer ethanol concentration."

The average total steam energy requirement of the unit during the two experiments was 3.88 megajoules per liter (13,900 Btu per gallon) of ethanol, exclusive of boiler efficiency.

Interrelationships of parameters in the distillation process were studied by use of correlation analyses among 22 system variables. For instance, atmospheric fluctuations have been found to correlate with performance of the distillation equipment for conditions of experiment two. Three percent of the measured ethanol in the corn-beer could not be accounted for in the distillation process. A study of the distillation interrelationship did not provide insight as to what the source of the difficulty was.

The total steam energy requirement of distillation was partitioned into four components. Heating of the corn-beer required 20 percent of the energy; reflux generation, 38 percent; product condensation, 18 percent; and system thermal losses absorbed the remaining 24 percent.

The distillation tower and associated equipment operated satisfactorily during the experimentation. Small-scale ethanol production from corn appears technically feasible based upon this study. However, additional experimentation is needed to establish long term equipment performance.

RECOMMENDATIONS FOR FURTHER RESEARCH

Further research is needed to fully characterize the performance of the South Dakota State University distillation unit. Three additional operating parameters, "corn-beer flow rate," "corn-beer inlet temperature," and "product ethanol composition" should each be independently studied for effects upon the "energy per liter ethanol" consumed. The two most important are "corn-beer flow rate" and "product ethanol concentration"; as Aries (1947) has studied "corn-beer inlet temperature."

In addition, various combinations of the variables should be studied for interaction effects. The quantity of ethanol extracted in the distillation process is somewhat less than that contained in the intitial corn-beer. The source of the loss should be determined. Also, work is needed in establishing a more accurate laboratory procedure for determining the "stillage residual ethanol concentration" associated with each "center stripper temperature" setting.

REFERENCES

1. Aries, E. R. 1947. Steam consumption of two-column alcohol distillation units. International Sugar Journal 49(578): 45-47.

2. Beckwith, T. G. and N. L. Buck. 1969. Mechanical measurements. Addison-Wesley Publishing Company, Reading, Massachusetts, 642 p.

3. Bossong, K. 1980. A history of alcohol fuels. The Energy Consumer. U. S. Department of Energy, Office of Consumer Affairs, U. S. Government Printing Office, Washington, D. C. January: 2-3.

4. Brachvogel, J. K. 1907. Industrial alcohol, its manufacture and uses. Munn & Company, Publishers, New York, 516 p.

5. Calingaert, G. 1925. Multiple-Effect distillation of ethyl alcohol. Chemical and Metallurgical Engineering 32(9): 362-363.

6. Campion, M. 1952. Fuel economy in the distillery. International Sugar Journal 54: 157.

7. Carney, T. P. 1949. Laboratory fractional distillation. The Macmillan Company, New York, 259 p.

8. Dobbs, T. L., R. Hoffman, and A. Lundeen. 1981. Framework for examining economic feasibility of small-scale alcohol plants. Series Number 81-3. Economics Department, South Dakota State University, 34 p.

9. Dobson, C. B. 1950. Industrial alcohol, present day distillation practice. Chemistry and Industry: 599-600.

10. Freshwater, D. C. 1951. Thermal economy in distillation. Transactions of the Institution of Chemical Engineers 29(1): 149-160.

11. Herman, A., E. M. Stallings, and H. F. Willkie. 1942. Chemical engineering developments in a grain distillery. American Institute of Chemical Engineers 38: 791-812.

12. Horwitz, W. 1970. Official methods of analysis of the association of official analytical chemists. Association of Official Analytical Chemists, Washington, D. C. 1015 p.

13. Jantzen, D. and T. McKinnon. 1980. Preliminary energy balance and economics of a farm-scale ethanol plant. Solar Energy Research Institute, Golden, Colorado, 9 p.

REFERENCES, continued

14. Jennings, B. H. 1970. Environmental engineering, analysis and practice. International Textbook Company, Scranton, Pennsylvania, 765 p.

15. Mariller, C. 1945. Determination of the steam consumption of a rectification column. Association des Chemistes 62: 291-299.

16. McCabe, W. L. and J. C. Smith. 1976. Unit operations of chemical engineering, 3rd ed. McGraw-Hill Book Company, New York, 1028 p.

17. Ogden, R. L. 1979. Production process for alcohol from foodstuffs. p 15-18. In: Cooperative Extension Service, University of Nebraska. Ethanol production and utilization, 83 p.

18. Oura, E. 1977. Reaction products of yeast fermentations. Process Biochemistry 12: 19-21.

19. Pennwalt Corporation. Sharples horizontal super-d-canter centifuges, bulletin 1270. Sharples-Stokes Division, Penwalt Corporation, Warminster, Pennsylvania, 13 p.

20. Physics Department. 1980. Physics 211 laboratory manual. South Dakota State University, Brookings, South Dakota, 60 p.

21. Prescott, S. C. and C. G. Dunn. 1959. Industrial microbiology, 3rd ed, McGraw-Hill Book Company, Incorporated, New York, 945 p.

22. Pyke, M. 1965. The manufacture of scotch grain whisky. Institute of Brewing 71: 209-218.

23. Reed, G. and H. J. Peppler. 1973. Yeast technology. The AVI Publishing Company, Incorporated, Westport, Connecticut, 378 p.

24. Reich, G. T. 1929. Ethyl alcohol industry improves its distillation process. Chemical and Metallurgical Engineering, 36(12): 716-719.

25. Spink, L.K. 1972. Principles and practice of flow meter engineering, 2nd ed. The Foxboro Company, Foxboro, Massachusetts, 575 p.

26. Suomalain, H. 1971. Yeast and its effect on the flavor of alcoholic beverages. Institute of Brewing, 77:164-177.

27. Underkofler, L.A., and Hickey, R.J. 1954. Industrial fermentations, Volume 1. Chemical Publishing Company, Incorporated, New York, 565 p.

REFERENCES, continued

28. United States Bureau of the Census. 1980. Statistical abstract of the United States. United States Government Printing Office, Washington, D.C., 1059 p.

29. Wall Street Journal. "Cash Prices." Wall Street Journal. 1973-1982.

30. Walpole, R.E., and R.H. Myers. 1978. Probability and statistics for engineers and scientists, 2nd ed. Mcmillan Publishing Company, Incorporated, New York, 580 p.

31. Weast, R.C. 1980. CRC handbook of chemistry and physics. CRC Press, Incorporated, Boca Raton, Florida, C1-C751.

32. Webb, A. D. 1963. Fusel oil. p 317-353. In: W. W. Umbreit (ed.). Applied microbiology. Academic Press, New York.

33. Westby, C.A., and W.R. Gibbons. 1982. Farm-scale production of fuel ethanol and wet grain from corn in a batch process. Biotechnology and Bioengineering; in press.

34. Yang, R.D., D.A. Grow, and W.E. Goldstein. 1982. Pilot plant studies of ethanol production. Gasohol USA 4(2): 13-18.

35. Gunness, R.C. and J.G. Baker. 1938. Performance of commercial perforated plate distillation column. American Institute of Chemical Engineers 34: 707-730.

APPENDIXES

APPENDIX A

PROJECT BACKGROUND

The alcohol fuels research project at South Dakota State University was initiated through the efforts of an industrial microbiologist in the Microbiology Department. Dr. Paul Middaugh had the initial insight to produce a research effort aimed at converting cellulosic agricultural residues and other waste materials into glucose, a simple sugar. The glucose could then be used for a number of purposes, including the production of ethyl alcohol (ethanol). In April of 1978, Middaugh received a $30,000 grant from the East River Electric Cooperative of Madison, South Dakota, to begin this study. By April of 1982, a total of five grants from various sources were awarded to Middaugh's project.

A small scale plant that could convert glucose to ethyl alcohol was established. Through fermentation, the glucose was converted to ethanol and then the ethanol extracted, refined, and finally utilized as fuel for internal combustion (gasoline) engines.

Dennis Vander Griend, a mechanical engineering student, was the key to the establishment of the ethanol extraction and refinement process. Through his efforts, a binary distillation tower was constructed of mild steel. The tower, a two-column unit of sieve tray design operating at atmospheric pressure, was 30.5 centimeters (12.0 inches) in diameter and capable of refining 83 liters (22 gallons) of 95 percent by volume ethyl alcohol per hour.

On April 19, 1979, Dr. Middaugh received notice that his application for a small scale, alcohol fuel producers permit from the United States Department of Treasury's Bureau of Alcohol, Tobacco, and Firearms, was approved.

Middaugh first publicly demonstrated the distillation tower at the Appropriate Community Technology (ACT) '79 Fair at the Washinton, D.C., Mall on April 27-30th, 1979. Operation of the tower reportedly drew large crowds including a number of Congressmen.

At this point, public interest in ethanol production was intense. With the skyrocketing price of gasoline, the near rationing conditions of the summer of 1979, and the relatively low prices for agricultural products, including corn (see Figure A-1); ethanol production became a promising dream for the farm community.

During this time, production research was being conducted at the glucose facility. Corn became the primary feedstock; the conversion of cellulose being a complex issue requiring much more time to master. But, by Spring of 1980, it was obvious that the mild

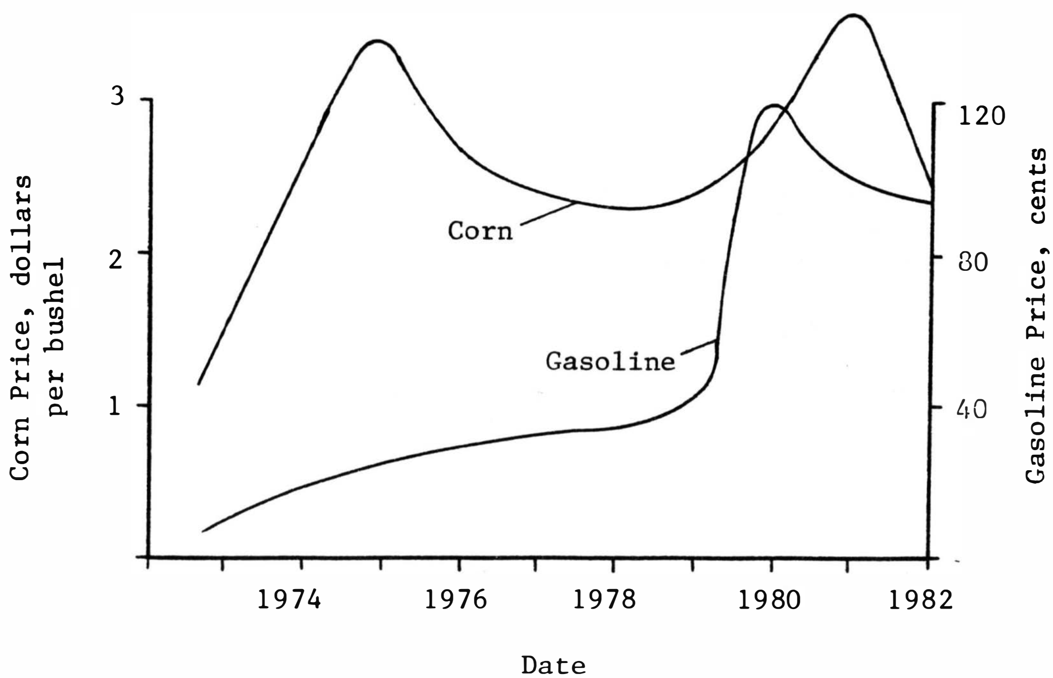


Figure A-1: Recent Corn and Gasoline Price Trends (Wall Street Journal)

steel tower needed to be replaced with stainless steel. Rusting of interior components clearly led to the alteration of design characteristics and blockage of some perforations in the rectification trays. Modifications in design of the mild steel tower were made and a new, stainless steel unit was installed. The new distillation tower was fabricated and loaned to SDSU by Arlon Industries, Inc., of Sheldon, Iowa. Arlon is a private distillation tower frabicator owned, in part, by Denny's brother, Dave Vander Griend. As of April, 1982, Arlon has fabricated a total of 14 distillation towers ranging from 30.5 to 88.9 centimeters (12 to 35 inches) in diameter.

Project leader Paul Middaugh resigned his position at SDSU in May of 1980. He is now employed with Northwest Pacific Energy Company, Inc., of Pasco, Washington. The SDSU project has since been reorganized. The project is now an interdepartmental effort pooling the resources and efforts of the Departments of Agricultural Engineering, Microbiology, Economics, Dairy Science, and Mechanical Engineering.

APPENDIX B

FIGURES AND TABLES

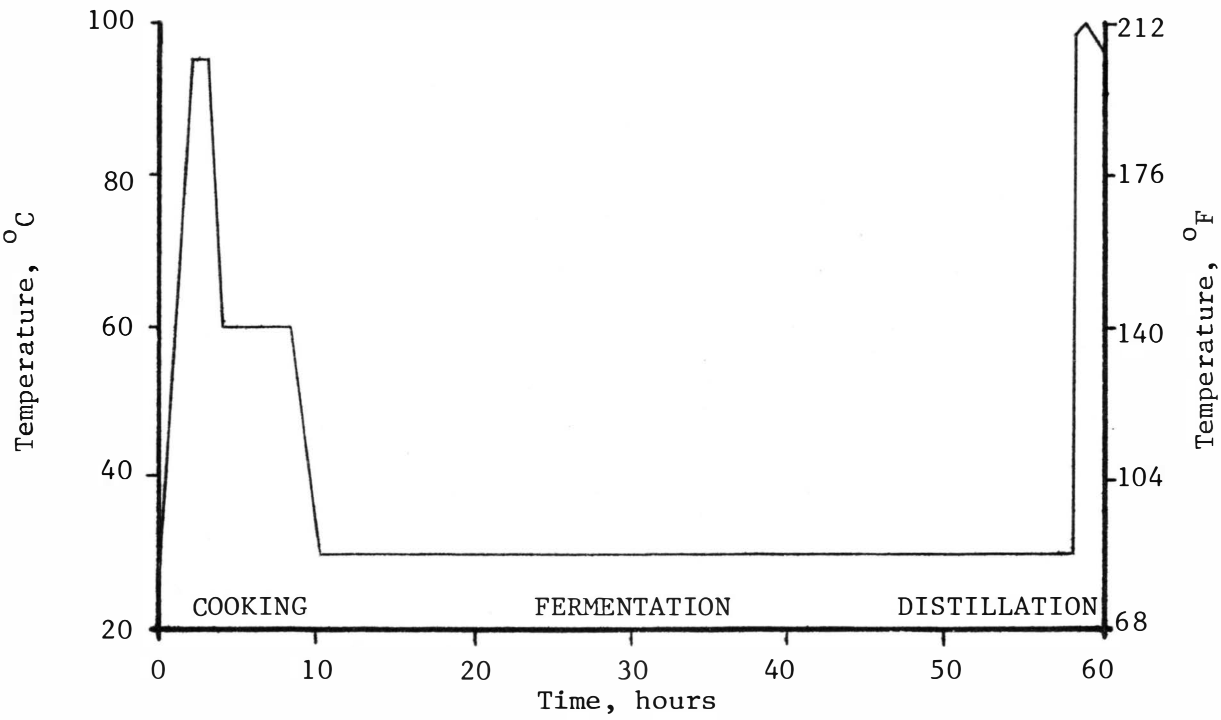


Figure B-1: Time-Temperature Diagram for the Corn-Mash and Corn-Beer Slurries During the Ethanol Production Process

Table B-1: Typical Sieve Analyses of Ground Corn

ASTM Sieve No.	Sieve Size	Percent, by weight, of material retained by sieve		
	(microns)	2.38mm* (3/32 inch) Screen	3.18mm (1/8 inch) Screen	4.48mm (3/16 inch) Screen
4	4760	0.0	0.3	0.0
6	3350	0.0	0.0	0.1
10	2000	0.1	1.3	5.0
20	850	27.4	36.2	39.6
40	420	42.8	34.3	28.1
60	250	20.6	14.5	16.0
80	180	6.7	9.9	8.3
100	149	1.2	1.8	1.7
140	105	0.8	1.4	0.6
Percent of material that passed through all the sieves=		0.3	0.8	0.3

*Employed for the Experimentation

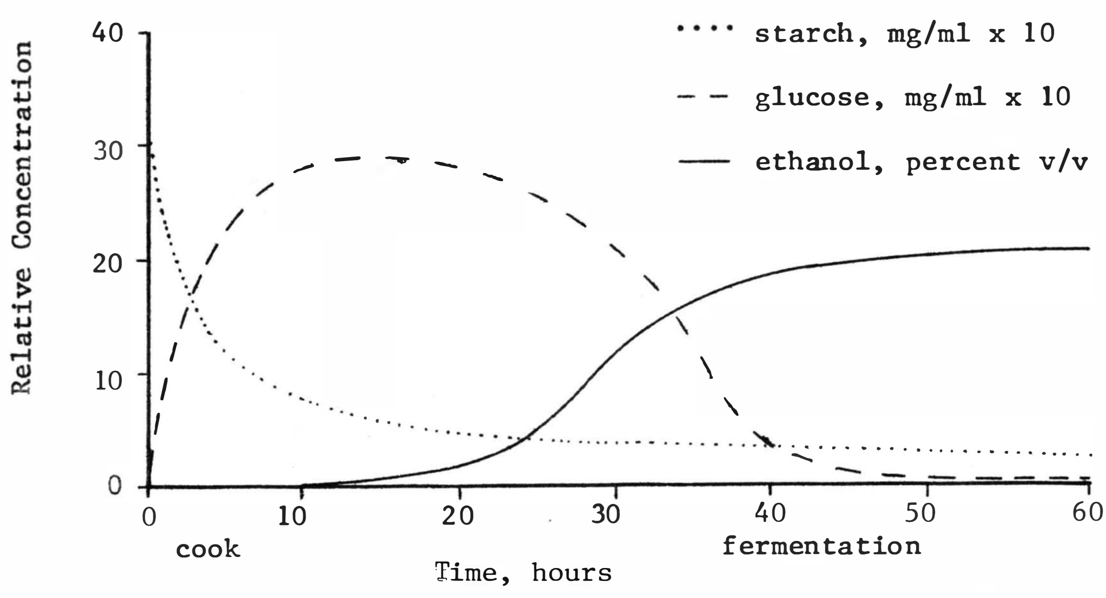


Figure B-2: Corn-Mash and Corn-Beer Component Changes During Cooking and Fermentation (Westby and Gibbons, 1982)

Table B-2: Proximate Analyses of Processed Materials in the Production of Ethanol From Corn[a]

	GROUND CORN	MOISTURE-FREE	CORN-MASH	MOISTURE-FREE	CORN-BEER	MOISTURE-FREE	STILLAGE	MOISTURE-FREE	WET GRAINS	MOISTURE-FREE	THIN STILLAGE	MOISTURE-FREE
	Figures in Percent by Weight											
Moisture[b]	11.5		76.5		89.6		92.5		67.7		97.3	
Crude Protein	8.7	9.8	2.2	9.5	2.5	24.0	1.9	26.1	10.1	31.4	0.36	13.0
Crude Fiber	2.1	2.4	0.75	3.2	0.76	6.6	0.52	6.7	3.0	9.1	0.006	0.21
Ether Extract (fat)	1.4	1.5	0.60	2.5	1.1	9.7	0.77	10.5	0.68	2.1	0.58	21.2
Ash	1.6	1.8	0.40	1.7	0.51	4.4	0.39	5.2	0.85	2.6	0.38	14.0
Nitrogen-free Extract	74.8	84.5	19.5	83.0	6.4	55.6	3.9	51.5	17.7	54.8	1.41	51.5
Total:	100.1	100.0	100.0	99.9	100.9	100.3	100.0	100.0	100.0	100.0	100.0	100.0
Suspended Solids			12.9		1.8		1.4				1.6	

[a] Average of three batches from three months during the summer of 1980. Analyses performed by Station Biochemistry, SDSU, Brookings, SD 57007.

[b] All figures are percent by weight of samples taken from batches started with fresh water. Moisture includes all volatiles, including ethanol.

TABLE B-3: Twenty-Two System Variables Employed for Correlation Studies of Distillation Interrelationships

VARIABLE	UNITS	DESCRIPTION
(1) Atmospheric Pressure	inches of mercury	On-Sight Pressure
(2) Corn-Beer Ethanol Concentration	percent by volume	Ethanol Portion of Corn-Water-Ethanol Mixture Before Distillation (Appendix G)
(3) Stillage Residual Ethanol Concentration	percent by volume	Ethanol Portion of Corn-Water-Ethanol Mixture After Distillation (Appendix G)
(4) Energy Flow Rate	megajoules per hour	Determined by Steam Measurement Device (Appendix F) Upon Inlet to Stripper
(5) Product Flow Rate	liters per hour	Measured by Bucket and Stop Watch
(6) Product Ethanol Concentration	percent by volume	Measured by Hydrometer (Appendix D)
(7) Product Anhydrous Ethanol Equivalent Flow Rate	liters per hour	Determined by (5) x (0.01) x (6)
(8) Energy Per Liter Ethanol	megajoules per liter	Determined by (4) / (7)
(9) Center Rectifier Temperature	degrees centigrade	Measured on Eleventh Tray from Bottom (Appendix E)
(10) Top Rectifier Temperature	degrees centigrade	Measured on Top Tray (Appendix E)
(11) Stillage Flow Rate	liters per hour	Measured by Bucket and Stop Watch
(12) Corn-Beer Flow Rate	liters per hour	Measured by Bucket and Stop Watch
(13) Corn-Beer Inlet Temperature	degrees centigrade	Measured at Inlet to Rectifier Preheater (Appendix E)
(14) Corn-Beer Outlet Temperature	degrees centigrade	Measured at Outlet from Rectifier Preheater (Appendix E)
(15) Reflux Flow Rate	liters per hour	Measured by Bucket and Stop Watch
(16) Reflux Temperature	degrees centigrade	Measured at Inlet to Top of Rectifier (Appendix E)
(17) Stillage Temperature	degrees centigrade	Measured at Outlet of Stripper (Appendix E)
(18) Water Flow Rate	liters per hour	Measured by Bucket and Stop Watch
(19) Water Inlet Temperature	degrees centigrade	Measured at Condenser Inlet (Appendix E)
(20) Water Outlet Temperature	degrees centigrade	Measured at Condenser Outlet (Appendix E)
(21) Center Stripper Temperature	degrees centigrade	Measured on Seventh Tray from Bottom (Appendix E)
(22) Apparent Ethanol Loss	percent of total	Determined by (12) x (0.01) x (2) - (7)

Table B-4: Raw Data for Experiments One and Two

Experiment	DATE OF DISTILLATION month-day	ATMOSPHERIC PRESSURE inches Hg	CORN-BEER ETHANOL CONCENTRATION, percent v/v	STILLAGE RESIDUAL ETHANOL CONCENTRATION, percent v/v	ENERGY FLOW RATE MJ/hr	PRODUCT FLOW RATE l/hr	PRODUCT ETHANOL CONCENTRATION percent v/v	PRODUCT ANHYDROUS ETHANOL EQUIVALENT FLOW RATE, l/hr	ENERGY PER LITER ETHANOL MJ/l	CENTER RECTIFIER TEMPERATURE degrees centigrade	TOP RECTIFIER TEMPERATURE degrees centigrade	STILLAGE FLOW RATE l/hr	CORN-BEER FLOW RATE l/hr	CORN-BEER INLET TEMPERATURE degrees centigrade	CORN-BEER OUTLET TEMPERATURE degrees centigrade	REFLUX FLOW RATE l/hr	REFLUX TEMPERATURE degrees centigrade	STILLAGE TEMPERATURE degrees centigrade	WATER FLOW RATE l/hr	WATER INLET TEMPERATURE degrees centigrade	WATER OUTLET TEMPERATURE degrees centigrade	CENTER STRIPPER TEMPERATURE degrees centigrade	APPARENT ETHANOL LOSS percent v/v
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
Experiment One	07-29	28.16	10.08	0.25	269.2	75.50	91.50	69.10	3.896	90.8	78.2	798	760	31.4	74.8	0.0	32.6	98.8	821	16.6	37.2	95.0	10.87
	08-03	28.18	10.14	0.33	257.6	80.70	92.00	74.20	3.472	88.7	77.9	781	733	33.5	74.1	1.5	33.1	97.5	727	17.7	41.7	94.4	0.17
	08-03	28.19	10.08	0.15	271.5	76.70	92.50	70.90	3.829	88.6	77.9	778	733	32.8	74.2	10.8	31.6	97.7	484	17.3	52.0	95.0	4.21
	08-05	28.25	10.49	0.00	266.4	77.70	93.00	72.30	3.685	85.7	77.8	856	731	33.9	74.9	12.0	29.1	96.6	810	17.1	39.7	95.0	6.06
	08-05	28.24	10.76	0.09	278.3	78.10	92.25	72.00	3.865	90.1	78.0	801	731	33.6	75.4	86.4	30.3	97.2	848	17.5	42.9	95.6	9.24
	08-12	28.19	10.48	0.46	264.7	76.40	92.25	70.50	3.755	91.9	77.8	757	739	34.5	75.4	0.0	29.8	100.5	772	17.1	46.1	94.4	9.85
	08-12	28.20	10.42	0.20	295.8	80.20	92.75	74.40	3.976	90.0	77.9	836	739	33.4	74.9	33.6	31.2	100.5	833	16.9	38.1	95.6	3.50
	08-20	28.25	9.86	0.05	279.5	77.10	94.00	72.50	3.855	85.6	77.9	847	749	34.5	74.8	24.0	28.9	101.4	795	17.6	43.0	95.6	1.86
	08-20	28.24	9.86	0.15	266.0	76.60	91.75	70.30	3.784	91.4	78.2	809	738	33.8	75.3	6.9	33.0	101.6	802	17.0	38.9	94.4	3.51
	09-03	28.44	10.06	0.20	276.9	79.00	92.25	72.90	3.798	90.7	78.3	838	750	30.9	75.1	6.9	27.8	100.6	490	15.3	52.0	95.0	3.50
	09-12	28.14	10.24	0.27	266.4	78.10	92.50	72.20	3.690	89.5	78.0	797	749	32.6	75.0	0.0	27.1	100.4	446	18.2	52.7	94.4	6.23
	09-12	28.14	10.39	0.20	296.5	81.00	93.25	75.50	3.927	92.1	77.8	817	752	32.1	74.8	28.8	29.8	100.7	794	15.6	44.7	95.6	3.49
	Ave.	28.22	10.24	0.20	274.1	78.09	92.50	72.23	3.794	89.6	78.0	810	742	33.1	74.9	17.6	32.0	99.5	719	17.0	44.1	95.0	5.23
Experiment Two	09-24	28.24	6.79	0.22	241.1	51.80	94.25	48.82	4.929	96.6	78.0	828	744	33.2	75.0	0	0	100.8	526	15.2	33.9	95.0	3.48
	09-24	28.29	10.06	0.14	266.8	74.40	92.50	68.82	3.877	91.6	78.1	814	748	30.7	74.6	0	0	101.1	544	14.6	43.2	95.0	9.34
	10-01	28.38	9.62	0.35	266.0	75.60	93.25	70.50	3.773	85.7	78.2	839	749	31.6	74.8	0	0	101.5	554	15.3	43.4	95.0	2.20
	10-08	28.00	13.78	0.27	291.2	99.99	90.75	93.56	3.112	90.5	78.2	780	743	29.7	73.9	0	0	101.4	822	14.4	41.6	95.0	9.43
	10-22	28.58	9.66	0.30	261.0	74.10	94.25	69.84	3.737	83.1	78.2	809	746	31.6	74.8	0	0	101.4	482	13.9	45.2	95.0	3.18
	10-22	28.64	12.90	0.30	286.8	98.80	93.00	91.88	3.121	85.5	78.4	821	732	29.2	74.2	0	0	101.8	653	13.7	45.5	95.0	2.77
	10-29	27.92	13.06	0.22	294.4	99.99	91.50	94.06	3.130	90.5	78.0	794	762	28.8	74.0	0	0	101.3	809	13.7	41.1	95.0	5.80
	10-29	27.86	10.29	0.22	274.3	79.20	92.00	72.86	3.764	91.4	77.7	792	745	29.3	73.6	0	0	100.9	790	13.7	35.1	95.0	5.22
	12-15	28.28	6.87	0.44	237.6	50.30	94.50	47.53	4.999	80.4	77.9	830	755	32.8	74.0	0	0	95.6	826	10.9	26.6	95.0	9.13
	12-15	28.28	13.09	0.25	283.6	92.70	93.50	86.67	3.272	81.8	78.0	809	730	27.5	74.0	0	0	96.2	781	11.1	35.7	95.0	10.25
	12-19	28.44	7.05	0.32	231.8	49.20	93.50	46.00	5.039	86.2	78.3	772	702	32.7	75.0	0	0	95.7	826	10.6	21.4	95.0	7.59
	12-19	28.34	6.86	0.35	238.8	51.20	93.50	47.87	4.989	85.0	78.1	784	731	32.5	74.6	0	0	95.5	812	10.7	21.8	95.0	4.76
	Ave.	28.27	10.00	0.28	274.5	74.77	93.04	69.87	3.979	86.5	78.1	806	740	30.8	74.4	0	0	99.4	702	13.2	36.1	95.0	6.11

Table B-5: Correlations Among 22 System Variables for Experiment One ('r' / PROB F)

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
1	1.00	-0.19	-0.31	0.02	-0.01	0.01	0.01	0.01	-0.12	0.52	0.48	-0.04	-0.22	0.28	0.07	-0.53	0.10	-0.21	-0.46	0.21	0.11	-0.19
	0.000	0.563	0.332	0.960	0.995	0.969	0.981	0.967	0.715	0.080	0.115	0.908	0.486	0.375	0.836	0.076	0.746	0.505	0.131	0.510	0.743	0.557
2	-0.19	1.00	0.05	0.24	0.27	0.03	0.25	0.11	0.15	-0.47	-0.10	-0.39	0.17	0.34	0.59	0.11	-0.45	0.30	0.04	-0.07	0.28	0.51
	0.563	0.000	0.882	0.458	0.392	0.925	0.438	0.742	0.640	0.120	0.762	0.210	0.606	0.280	0.043	0.724	0.138	0.348	0.898	0.828	0.385	0.090
3	-0.31	0.05	1.00	-0.28	0.06	-0.47	-0.09	-0.27	0.62	-0.03	-0.75	0.18	-0.09	0.01	-0.45	0.79	0.23	-0.17	0.01	0.20	-0.57	0.19
	0.-32	0.882	0.000	0.385	0.860	0.124	0.792	0.389	0.033	0.935	0.005	0.565	0.770	0.989	0.138	0.002	0.475	0.596	0.999	0.540	0.052	0.546
4	0.02	0.24	-0.28	1.00	0.47	0.51	0.57	0.80	0.19	-0.16	0.46	0.27	-0.20	0.12	0.52	-0.48	0.35	0.23	-0.49	-0.08	0.84	-0.13
	0.960	0.458	0.385	0.000	0.122	0.090	0.054	0.002	0.553	0.620	0.136	0.393	0.528	0.508	0.084	0.117	0.267	0.467	0.106	0.796	0.001	0.680
5	-0.01	0.27	0.06	0.47	1.00	0.27	0.96	-0.12	0.09	-0.28	0.23	-0.10	-0.15	-0.28	0.23	-0.30	0.01	0.01	-0.25	0.04	0.27	-0.57
	0.995	0.392	0.860	0.122	0.000	0.398	0.001	0.713	0.773	0.380	0.474	0.755	0.631	0.373	0.472	0.348	0.992	0.993	0.427	0.907	0.390	0.051
6	0.01	0.03	-0.47	0.51	0.27	1.00	0.53	0.24	-0.56	-0.58	0.55	0.04	0.33	-0.11	0.23	0.39	0.20	0.08	0.03	0.12	0.59	-0.38
	0.969	0.925	0.124	0.090	0.398	0.000	0.079	0.458	0.058	0.047	0.061	0.907	0.292	0.745	0.469	0.204	0.525	0.811	0.922	0.716	0.045	0.222
7	0.01	0.25	-0.09	0.57	0.96	0.53	1.00	-0.03	-0.08	-0.41	0.37	-0.07	-0.04	-0.27	0.27	-0.38	0.07	0.03	-0.23	0.06	0.41	-0.62
	0.981	0.438	0.792	0.054	0.001	0.079	0.000	0.924	0.815	0.185	0.234	0.824	0.894	0.390	0.404	0.223	0.832	0.919	0.481	0.859	0.183	0.033
8	0.01	0.11	-0.27	0.80	-0.12	0.24	-0.03	1.00	0.28	0.11	0.29	0.39	-0.22	0.34	0.44	-0.31	0.37	0.26	-0.43	-0.15	0.73	0.28
	0.967	0.742	0.390	0.002	0.713	0.458	0.924	0.000	0.370	0.741	0.368	0.216	0.492	0.283	0.152	0.326	0.329	0.410	0.161	0.644	0.007	0.376
9	-0.12	0.15	0.62	0.19	0.09	-0.56	-0.08	0.28	1.00	0.32	-0.49	0.29	-0.40	0.39	-0.01	0.39	0.38	0.01	-0.47	0.07	-0.18	0.32
	0.715	0.640	0.033	0.553	0.773	0.058	0.815	0.370	0.000	0.310	0.104	0.363	0.201	0.213	0.981	0.216	0.226	0.976	0.125	0.831	0.568	0.311
10	0.52	-0.47	-0.03	-0.16	-0.28	-0.58	-0.41	0.11	0.22	1.00	0.10	0.41	-0.59	0.25	-0.14	-0.81	0.25	-0.25	-0.32	0.04	-0.19	0.09
	0.080	0.120	0.935	0.620	0.380	0.049	0.185	0.741	0.310	0.000	0.766	0.189	0.043	0.425	0.667	0.585	0.440	0.428	0.311	0.891	0.561	0.788
11	0.48	-0.10	-0.75	0.46	0.23	0.55	0.37	0.29	-0.49	0.10	1.00	0.15	-0.07	0.11	0.20	-0.77	0.13	0.19	-0.30	-0.26	0.55	-0.33
	0.115	0.762	0.005	0.136	0.474	0.061	0.234	0.368	0.104	0.766	0.000	0.648	0.829	0.731	0.537	0.003	0.677	0.551	0.341	0.421	0.681	0.302
12	-0.04	-0.39	0.18	0.37	-0.10	0.04	-0.07	0.39	0.29	0.21	0.15	1.00	-0.61	0.08	-0.31	-0.03	0.55	-0.11	-0.43	0.07	0.13	0.14
	0.908	0.210	0.565	0.393	0.755	0.907	0.824	0.216	0.363	0.189	0.648	0.000	0.034	0.794	0.326	0.924	0.061	0.724	0.167	0.825	0.681	0.660
13	-0.22	0.17	-0.09	-0.20	-0.15	0.33	-0.04	-0.22	-0.40	-0.59	-0.07	-0.61	1.00	0.15	0.19	0.28	-0.03	0.46	0.63	-0.31	-0.37	-0.08
	0.486	0.606	0.770	0.528	0.631	0.292	0.894	0.492	0.201	0.043	0.829	0.034	0.000	0.653	0.561	0.381	0.914	0.136	0.028	0.320	0.819	0.810
14	0.28	0.34	0.01	0.12	-0.28	-0.11	-0.27	0.34	0.39	0.25	0.11	0.08	0.15	1.00	0.31	0.19	0.39	0.27	-0.15	-0.08	0.03	0.55
	0.375	0.280	0.989	0.706	0.373	0.745	0.390	0.283	0.213	0.425	0.731	0.794	0.653	0.000	0.320	0.556	0.215	0.400	0.640	0.796	0.936	0.063
15	0.07	0.59	-0.45	0.52	0.23	0.23	0.27	0.44	-0.01	-0.14	0.20	-0.31	0.19	0.31	1.00	-0.40	-0.23	0.41	0.04	-0.17	0.71	0.17
	0.836	0.043	0.138	0.084	0.472	0.469	0.404	0.152	0.981	0.667	0.537	0.326	0.561	0.320	0.000	0.199	0.470	0.190	0.897	0.587	0.009	0.603
16	-0.53	0.11	0.79	-0.48	-0.30	-0.40	-0.38	-0.31	0.39	-0.18	-0.77	-0.03	0.28	0.19	-0.40	1.00	0.16	-0.13	0.46	0.15	-0.70	0.40
	0.076	0.724	0.002	0.117	0.347	0.204	0.223	0.326	0.216	0.585	0.003	0.924	0.381	0.556	0.199	0.000	0.618	0.691	0.136	0.633	0.012	0.202
17	0.10	-0.45	0.23	0.35	0.01	0.20	0.07	0.37	0.38	0.25	0.13	0.55	-0.03	0.39	-0.23	0.16	1.00	-0.05	-0.27	0.10	-0.01	-0.21
	0.746	0.138	0.475	0.267	0.992	0.525	0.832	0.239	0.226	0.440	0.677	0.061	0.914	0.215	0.470	0.618	0.000	0.878	0.394	0.754	0.971	0.503
18	-0.21	0.30	-0.17	0.23	0.01	0.08	0.03	0.26	0.01	-0.25	0.19	-0.11	0.46	0.27	0.41	-0.13	-0.05	1.00	-0.03	-0.90	0.37	0.18
	0.505	0.348	0.596	0.467	0.993	0.811	0.919	0.410	0.976	0.428	0.551	0.724	0.136	0.400	0.190	0.691	0.878	0.000	0.936	0.001	0.238	0.570
19	-0.46	0.04	0.01	-0.49	-0.25	0.03	-0.23	-0.43	-0.47	-0.32	-0.30	-0.43	0.63	-0.15	0.04	0.46	-0.27	-0.03	1.00	-0.03	-0.31	0.03
	0.131	0.898	0.999	0.106	0.427	0.922	0.481	0.161	0.125	0.311	0.341	0.167	0.028	0.640	0.897	0.136	0.394	0.936	0.000	0.933	0.333	0.914
20	0.21	-0.07	0.20	-0.08	0.04	0.12	0.06	-0.15	0.07	0.04	-0.26	0.07	-0.31	-0.08	-0.17	0.15	0.10	-0.90	-0.28	1.00	-0.20	-0.08
	0.510	0.818	0.540	0.796	0.907	0.716	0.859	0.644	0.831	0.891	0.421	0.825	0.320	0.796	0.587	0.633	0.754	0.001	0.933	0.000	0.523	0.816
21	0.11	0.28	-0.57	0.48	0.27	0.59	0.41	0.73	-.018	-0.19	0.55	0.13	-0.07	0.03	0.71	-0.70	-0.01	0.37	-0.31	-0.20	1.00	
	0.743	0.385	0.052	0.001	0.390	0.045	0.183	0.007	0.568	0.561	0.062	0.681	0.819	0.936	0.009	0.012	0.971	0.238	0.333	0.523	0.000	
22	-0.19	0.51	0.19	-0.13	-0.57	-0.38	-0.62	0.28	0.32	0.09	-0.33	0.14	-0.08	0.55	0.17	0.40	-0.21	0.18	0.03	-0.08		1.00
	0.557	0.090	0.546	0.680	0.051	0.222	0.033	0.376	0.311	0.788	0.302	0.660	0.801	0.063	0.603	0.202	0.503	0.570	0.914	0.816		0.000

Table B-6: Correlations Among 22 System Variables for Experiment Two ('r' / PROB F)

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
1	1.00 0.000	-0.26 0.405	0.39 0.215	-0.35 0.263	-0.26 0.414	0.67 0.018	-0.26 0.419	0.20 0.530	-0.61 0.034	0.72 0.009	0.34 0.280	-0.43 0.163	0.34 0.274	0.62 0.031	0.00 1.000	0.00 1.000	-0.15 0.652	-0.78 0.118	-0.16 0.621	0.05 0.886	0.00 1.000	-0.45 0.139
2	-0.26 0.405	1.00 0.000	-0.41 0.189	0.98 0.001	0.99 0.001	-0.70 0.011	0.99 0.001	-0.97 0.001	0.33 0.290	0.14 0.657	-0.12 0.715	0.22 0.490	-0.92 0.001	-0.60 0.041	0.00 1.000	0.00 1.000	0.49 0.105	0.12 0.710	0.31 0.330	0.70 0.011	0.00 1.000	0.63 0.029
3	0.39 0.214	-0.41 0.189	1.00 0.000	-0.46 0.131	-0.41 0.189	0.48 0.110	-0.40 0.201	0.42 0.176	-0.73 0.008	0.18 0.580	0.15 0.632	-0.14 0.665	0.46 0.136	0.80 0.816	0.00 1.000	0.00 1.000	-0.52 0.082	0.28 0.373	-0.51 0.091	-0.40 0.109	0.00 1.000	-0.35 0.259
4	-0.35 0.263	0.98 0.001	-0.46 0.131	1.00 0.000	0.99 0.001	-0.73 0.007	0.99 0.001	-0.98 0.001	0.42 0.172	0.03 0.920	-0.04 0.897	0.37 0.241	-0.91 0.001	-0.62 0.031	0.00 1.000	0.00 1.000	0.60 0.039	0.05 0.874	0.43 0.165	0.76 0.004	0.00 1.000	0.54 0.072
5	-0.26 0.414	0.99 0.001	-0.41 0.189	0.99 0.001	1.00 0.000	-0.70 0.011	0.99 0.001	-0.99 0.001	0.36 0.248	0.14 0.672	-0.06 0.853	0.29 0.357	-0.90 0.001	-0.58 0.047	0.00 1.000	0.00 1.000	0.58 0.050	0.05 0.867	0.39 0.213	0.76 0.004	0.00 1.000	0.53 0.077
6	0.67 0.018	-0.70 0.011	0.48 0.110	-0.73 0.007	-0.70 0.011	1.00 0.000	-0.69 0.013	0.65 0.023	-0.82 0.001	0.04 0.893	0.49 0.103	-0.20 0.523	0.60 0.078	0.56 0.061	0.00 1.000	0.00 1.000	-0.46 0.132	-0.36 0.255	-0.34 0.281	-0.40 0.203	0.00 1.000	-0.55 0.066
7	-0.26 0.419	0.99 0.001	-0.40 0.201	0.99 0.001	0.99 0.001	-0.69 0.013	1.00 0.000	-0.99 0.001	0.35 0.266	0.14 0.660	-0.06 0.858	0.30 0.346	-0.89 0.001	-0.58 0.049	0.00 1.000	0.00 1.000	0.57 0.051	0.05 0.866	0.38 0.217	0.76 0.004	0.00 1.000	0.53 0.076
8	0.20 0.530	-0.97 0.001	0.42 0.167	-0.98 0.001	-0.99 0.001	0.65 0.023	-0.99 0.001	1.00 0.000	-0.34 0.280	-0.14 0.658	-0.01 0.997	-0.32 0.317	0.88 0.001	0.53 0.079	0.00 1.000	0.00 1.000	-0.62 0.030	0.06 0.854	-0.44 0.151	-0.83 0.001	0.00 1.000	-0.49 0.109
9	-0.61 0.034	0.33 0.290	-0.73 0.008	0.42 0.172	0.36 0.248	-0.82 0.001	0.35 0.266	-0.34 0.280	1.00 0.000	-0.14 0.675	-0.39 0.214	0.20 0.540	-0.28 0.382	-0.20 0.512	0.00 1.000	0.00 1.000	0.58 0.048	0.02 0.957	0.54 0.070	0.35 0.262	0.00 1.000	0.22 0.499
10	0.72 0.009	0.14 0.657	0.18 0.580	0.03 0.920	0.14 0.672	0.04 0.893	0.14 0.660	-0.14 0.658	-0.14 0.675	1.00 0.000	-0.04 0.901	-0.46 0.131	0.11 0.725	0.51 0.089	0.00 1.000	0.00 1.000	0.13 0.684	-0.26 0.407	0.07 0.839	0.27 0.396	0.00 1.000	-0.16 0.627
11	0.34 0.279	-0.12 0.715	0.15 0.632	-0.04 0.897	-0.06 0.853	0.49 0.103	-0.06 0.858	-0.01 0.997	-0.39 0.214	-0.04 0.901	1.00 0.000	0.48 0.015	0.15 0.642	0.17 0.592	0.00 1.000	0.00 1.000	0.26 0.409	-0.62 0.031	0.40 0.195	0.33 0.299	0.00 1.000	-0.33 0.300
12	-0.43 0.163	0.22 0.490	-0.14 0.665	0.37 0.241	0.29 0.357	-0.20 0.523	0.30 0.346	-0.32 0.317	0.20 0.540	-0.46 0.131	0.48 0.115	1.00 0.000	-0.14 0.656	-0.36 0.253	0.00 1.000	0.00 1.000	0.51 0.091	-0.24 0.448	0.53 0.074	0.45 0.145	0.00 1.000	0.05 0.868
13	0.34 0.274	-0.92 0.001	0.46 0.136	-0.91 0.001	-0.90 0.001	0.60 0.038	-0.89 0.001	0.88 0.001	-0.28 0.382	0.11 0.725	0.15 0.642	-0.14 0.656	1.00 0.000	0.70 0.011	0.00 1.000	0.00 1.000	-0.29 0.361	-0.24 0.448	-0.10 0.749	-0.52 0.082	0.00 1.000	-0.64 0.025
14	0.62 0.031	-0.60 0.041	0.08 0.816	-0.62 0.031	-0.58 0.047	0.56 0.061	-0.58 0.049	0.53 0.079	-0.20 0.523	0.51 0.089	0.17 0.592	-0.36 0.253	0.70 0.011	1.00 0.000	0.00 1.000	0.00 1.000	-0.06 0.862	-0.59 0.042	0.10 0.748	-0.11 0.745	0.00 1.000	-0.57 0.053
15	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000
16	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000
17	-0.15 0.652	0.49 0.105	-0.52 0.082	0.60 0.039	0.58 0.050	-0.46 0.132	0.57 0.051	-0.62 0.030	0.58 0.048	0.13 0.684	0.26 0.409	0.51 0.091	-0.29 0.361	-0.06 0.862	0.00 1.000	0.00 1.000	1.00 0.000	-0.58 0.048	0.94 0.001	0.89 0.001	0.00 1.000	-0.10 0.764
18	-0.48 0.118	0.12 0.710	0.28 0.373	0.05 0.874	0.05 0.867	-0.36 0.255	0.05 0.866	0.06 0.854	0.02 0.957	-0.26 0.407	-0.62 0.031	-0.24 0.448	-0.24 0.448	-0.59 0.042	0.00 1.000	0.00 1.000	-0.58 0.048	1.00 0.000	-0.68 0.015	-0.59 0.044	0.00 1.000	0.44 0.153
19	-0.16 0.621	0.31 0.330	-0.51 0.091	0.43 0.165	0.39 0.213	-0.34 0.281	0.38 0.217	-0.44 0.151	0.54 0.070	0.77 0.839	0.40 0.195	0.53 0.074	-0.10 0.749	0.10 0.748	0.00 1.000	0.00 1.000	0.94 0.001	-0.68 0.015	1.00 0.000	0.79 0.002	0.00 1.000	-0.14 0.668
20	0.05 0.886	0.70 0.011	-0.49 0.109	0.76 0.004	0.76 0.004	-0.40 0.203	0.76 0.004	-0.83 0.001	0.35 0.262	0.27 0.396	0.33 0.299	0.45 0.145	-0.52 0.082	-0.11 0.745	0.00 1.000	0.00 1.000	0.89 0.001	-0.59 0.044	0.79 0.002	1.00 0.000	0.00 1.000	0.13 0.692
21	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000	0.00 1.000
22	-0.45 0.139	0.63 0.029	-0.35 0.259	0.54 0.072	0.53 0.077	-0.55 0.066	0.53 0.076	-0.49 0.109	0.22 0.499	-0.16 0.627	-0.33 0.300	0.05 0.868	-0.64 0.025	-0.57 0.053	0.00 1.000	0.00 1.000	-0.10 0.764	0.44 0.153	-0.14 0.668	0.13 0.692	0.00 1.000	1.00 0.000

Table B-7: Correlations Among Components of Total Steam Energy Partitioning for Experiments One and Two

Experiment 1

	A	B	C	D
A	1.00 0.000	-0.34 0.278	0.48 0.117	-0.29 0.364
B	-0.34 0.278	1.00 0.000	-0.89 0.001	0.36 0.255
C	0.48 0.117	-0.89 0.001	1.00 0.000	-0.62 0.030
D	-0.29 0.364	0.36 0.255	-0.62 0.030	1.00 0.000

Experiment 2

	A	B	C	D
A	1.00 0.000	0.90 0.001	-0.73 0.007	0.76 0.004
B	0.90 0.001	1.00 0.000	-0.79 0.002	0.96 0.001
C	-0.73 0.007	-0.79 0.002	1.00 0.000	-0.78 0.003
D	0.76 0.004	0.96 0.001	-0.78 0.003	1.00 0.000

A: Corn-Beer Heating

B: Reflux Generation

C: Product Condensation

D: System Thermal Losses

Table B-8: Correlations Between 22 System Variables and the Components of Total Steam Energy Partitioning for Experiments One and Two

	Experiment One					Experiment Two			
	Component:					Component:			
	A	B	C	D		A	B	C	D
Variable: 1	0.03	0.11	-0.09	-0.05	1	0.14	0.21	-0.53	0.25
	0.932	0.723	0.780	0.866		0.663	0.509	0.076	0.433
2	-0.65	0.54	-0.46	0.15	2	-0.94	-0.97	0.80	-0.90
	0.022	0.067	0.137	0.616		0.001	0.001	0.002	0.001
3	0.27	-0.44	0.54	-0.53	3	0.24	0.42	-0.28	0.47
	0.388	0.157	0.067	0.079		0.453	0.169	0.376	0.126
4	0.15	0.50	-0.43	0.55	4	-0.88	-0.98	0.83	-0.95
	0.650	0.097	0.165	0.065		0.001	0.001	0.001	0.001
5	-0.28	0.10	-0.25	0.26	5	-0.91	-0.99	0.81	-0.94
	0.385	0.757	0.426	0.421		0.001	0.001	0.002	0.001
6	0.04	0.11	-0.13	0.23	6	0.60	0.67	-0.82	0.62
	0.905	0.730	0.698	0.474		0.039	0.017	0.001	0.032
7	-0.23	0.12	0.25	0.29	7	-0.91	-0.99	0.80	-0.94
	0.479	0.718	0.426	0.357		0.001	0.001	0.002	0.001
8	0.34	0.53	-0.34	0.46	8	0.90	0.99	-0.79	0.97
	0.281	0.079	0.289	0.135		0.001	0.001	0.002	0.001
9	0.34	0.09	0.14	-0.28	9	-0.16	-0.37	0.52	-0.40
	0.275	0.778	0.670	0.386		0.610	0.240	0.086	0.192
10	0.38	0.04	-0.02	-0.09	10	-0.20	-0.15	-0.12	-0.06
	0.218	0.900	0.941	0.773		0.533	0.632	0.701	0.863
11	0.01	0.18	-0.27	0.43	11	0.23	0.02	-0.18	-0.14
	0.999	0.584	0.389	0.160		0.472	0.943	0.582	0.665
12	0.69	-0.16	0.34	-0.21	12	-0.03	-0.30	0.41	-0.49
	0.013	0.619	0.283	0.523		0.936	0.350	0.187	0.103
13	-0.15	0.03	-0.06	-0.10	13	0.92	0.88	-0.73	0.78
	0.644	0.933	0.845	0.768		0.001	0.001	0.007	0.003
14	0.25	0.40	-0.15	-0.26	14	0.57	0.52	-0.76	0.49
	0.433	0.200	0.653	0.408		0.051	0.080	0.004	0.104
15	-0.43	0.97	-0.91	0.40	15	0.00	0.00	0.00	0.00
	0.164	0.001	0.001	0.198		1.000	1.000	1.000	1.000
16	0.23	-0.39	0.46	-0.47	16	0.00	0.00	0.00	0.00
	0.464	0.206	0.136	0.124		1.000	1.000	1.000	1.000
17	0.92	-0.20	0.32	-0.22	17	-0.23	-0.63	0.46	-0.75
	0.001	0.538	0.310	0.491		0.465	0.027	0.137	0.003
18	-0.13	0.36	-0.21	0.14	18	-0.27	0.05	0.31	0.21
	0.686	0.253	0.511	0.964		0.403	0.880	0.332	0.515
19	-0.21	-0.04	-0.17	0.05	19	-0.02	-0.45	0.28	-0.62
	0.505	0.895	0.588	0.884		0.944	0.142	0.378	0.032
20	0.10	-0.15	0.14	-0.16	20	-0.53	-0.82	0.52	-0.90
	0.758	0.652	0.669	0.620		0.075	0.001	0.084	0.001
21	0.00	0.00	0.00	0.00	21	0.00	0.00	0.00	0.00
	1.000	1.000	1.000	1.000		1.000	1.000	1.000	1.000
22	-0.09	0.31	-0.05	-0.18	22	-0.63	-0.48	0.61	-0.39
	0.777	0.326	0.886	0.573		0.029	0.112	0.036	0.209

SDSU ALCOLOL FUEL DATA SHEET, page two.
raw data in pencil, remainder in ink.

BATCH #:__________ DATE:__________ RESEARCHER:____________________

	TIME:
SYSTEM TEMPERATURES:	
2	HE EX BOT OUT
	HE EX TOP OUT
	HE EX BOT IN
5	HE EX TOP IN
36	BEER IN RECT
7	BEER OUT RECT
37	BEER IN STRIP
33	CENTER STRIP
10	TOP STRIP
11	STILLAGE
12	REFLUX
13	COOL H_2O IN
14	HOT H_2O OUT
	STEAM HEATER
16	STEAM
34	PRODUCT
38	RECT #1
21	RECT #2
22	RECT #3
23	RECT #4
24	RECT #5
25	RECT CENTER
26	RECT TOP
28	#1, STEAM OUT
30	#1, STEAM IN
SK3	#1, TOP
31	#2, STEAM OUT
32	#2, STEAM IN
SK2	#1, BOTTOM
29	#2, TOP
27	#2, BOTTOM
SK1	RED FERMENTER

Figure B-3: Temperature Data Form

SDSU ALCOHOL FUEL DATA SHEET, page one.
raw data in pencil, remainder in ink.

BATCH #:__________ DATE:__________ RESEARCHER:__________________

% BACKSET:________ CORN/WATER RATIO:__________ TOTAL BU:________

BEER % ETOH:______ ADDED WATER OR ETOH:__________ YIELD:________

TOTAL LITERS ETOH:_______ PERCENT ALCOHOL:_______

TOTAL WET GRAIN:_________ PERCENT MOISTURE:______

TIME:										
SYSTEM FLOW RATES:										
STEAM										
FEED C/S										
BUCKET TEST										
REFLUX #1 C/S										
REFLUX #2 C/S										
BUCKET TEST										
STILLAGE C/S										
BUCKET TEST										
EFFLUENT										
WET GRAIN										
COOLING WATER										
PRODUCT										
PRODUCT % ETOH:										
SAMPLE TEMP										
CORRECTED % ETOH										
STEAM PRESSURE:										
HYDROLICS PRES:										
ATMOSPHERIC PRES:										
ELECTRICAL: 3Ø										
1Ø										
MILL CURRENT										
HEATER CURRENT										
STILLAGE % ETOH:										

Figure B-4: Miscellaneous Data Form

APPENDIX C

EQUIPMENT

Pumps

The pumps employed at South Dakota State University will be described herein. Each pump is of positive displacement capability, and has hydraulic drive for infinite speed control. Operating performance are evaluated and recommendations made for the future. These particular pumps have been employed for a very limited period, approximately eight hours a week for one year.

Feed Pump

The pump employed to move corn-beer from the fermenters to the distillation tower is termed the feed pump (see Figure C-1). This pump moves a $30^{o}C$ ($86^{o}F$), aqueous solution of ethanol (10 percent by volume) and corn particles (8.5 percent by weight). The pumping rate ranges from 600 to 900 liters per hour (160 to 240 gallons per hour). The pump is also capable of moving tap water through the distillation

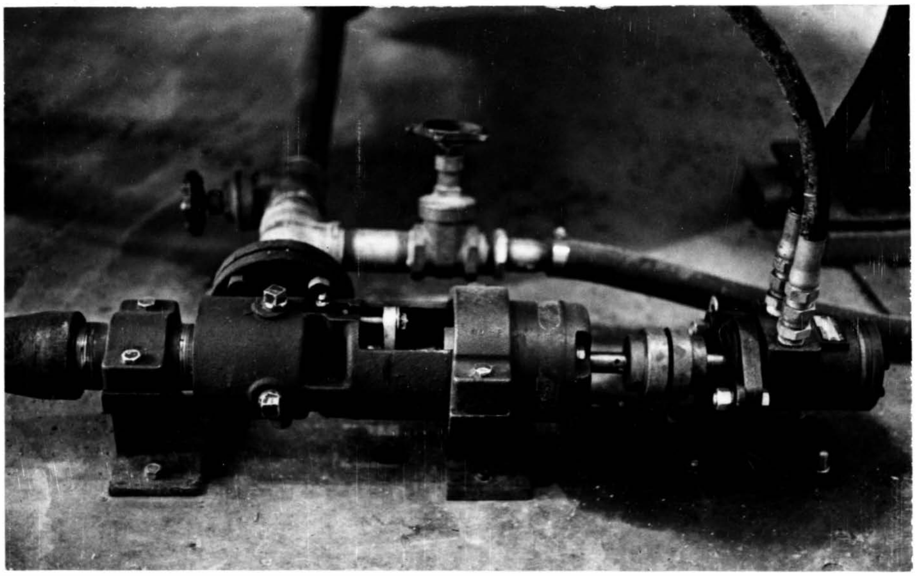


Figure C-1: Feed Pump

column for cleaning purposes.

SDSU employs a positive displacement, progressive cavity-type pump for this application. The pump is capable of moving the feed at a steady rate despite fluctuations in inlet and outlet pressures. This is important in maintaining a constant temperature within $\pm$ 0.2^{o}C (0.4^{o}F) in the stripper column.

The feed pump is made by Roper Pump Company, Commerce, Georgia 30529. The description on the pump is Figure Number 71202-GH2, type 1, Serial Number Y3132. The approximate retail cost is $665, including the hydraulic drive motor (Dobbs, 1982).

The Roper pump has performed its function adequately over the short test period of 400 hours of intermittant operation. The feed rate can be held steady after the first hour of operation of the pump.

It has been found that rapid deterioration of the stator may occur when this pump is accidently operated without fluid. All precautions should be taken to keep this from occuring and replacement stators should be kept in stock. In addition, due to the limited lubrication properties of the fluid stream, periodic replacement of the stator, and possibly rotor, can be anticipated.

Stillage Pump

The pump employed for continuous removal of spent corn-beer (stillage) from the stipper column is the same type as that employed for feeding the column. The Roper Figure Number 71202-GNC, Type 1, Spec 9082, Serial Number Y3304, progressive cavity-type pump is one size larger than that employed to feed the column (see Figure C-2). The stillage pump employed at SDSU is made to withstand a fluid temperature of 100°C (212°F) pumped at a rate of 700 to 1000 liters per hour (180 to 260 gallons per hour). The approximate cost of this pump and drive motor is $1030 (Dobbs, 1982).

The Roper pump in this application has also performed well during the short duration of the test. Once again, due to the limited lubrication properties of the fluid stream, periodic replacement of the stator, and possibly rotor, is anticipated.

Rectifier Pump

The pump employed to continuously move liquid water and alcohol from the bottom of the rectifier column to the top of the stripper column is called the rectifier pump. The purpose of the pump is to maintain the series connection of fluid flow between the columns.

The pump employed for this application is a positive displacement, gear-type pump. The pump is manufactured by Viking Pump Company, Cedar Rapids, Iowa 50613. The specific pump employed at SDSU is shown in Figure C-3. It's retail value is $325, including the hydraulic drive motor (Dobbs, 1982).

In order to reduce wear, this pump is oversized and operated at less than 200 revolutions per minute. Model HV 2341, Serial Number 1757853, is rated at approximately 230 liters per hour (60 gallons per hour) at this speed.

Again, the pump has proven to work properly in this application, but has shown some wear problems. Even teflon impregnated parts appear to wear in the water-ethanol mixture and need to be replaced periodically. The 400 hours of use has resulted in replacement of two interior parts.

Centrifuge

The centrifuge at South Dakota State University is employed to remove as many solids from the corn stillage as possible. The high moisture solids are fed to cattle as distillers wet grain while a portion of the liquid waste is returned to the next cook batch. The purpose of recycling the liquid is to boost the alcohol yield in the conversion process.

The make of the centrifuge is Sharples-Stokes Division of Pennwalt Corporation, 955 Mearns Road, Warminster, Pennsylvania 18974. It is a type BM.PF956/7482113, Serial Number 73-P600-187; capable of separating up to three-fourths of the solids from the stillage. See

Figure C-2: Stillage Pump

Figure C-3: Rectifier Pump

Table B-2 in Appendix B for analyses of the stillage, animal feed (distillers wet grain), and waste water (thin stillage). Figure C-4 is a photograph of the centrifuge. Figure C-5 shows the interior construction of the machine. The approximate cost of this piece of machinery is $32,000 (Hoffman, 1982).

Distillation Tower

The distillation tower pictured in Figure C-6 is a two-column unit of sieve tray (perforated plate) design. It is constructed of welded stainless steel and operates at atmospheric pressure. The columns are each 30.5 centimeters (12 inches) in diameter and 483 centimeters (190 inches) tall. The tower is capable of refining 90 liters (24 gallons) per hour of 92.5 percent ethanol product from a 10 percent ethanol corn-beer. The unit is on loan from Arlon Industries, Inc., P.O. Box 347, Sheldon, Iowa 51201. The approximate cost is $19,000 (Dobbs, 1982).

Figures C-7 through C-10 are scaled drawings of the apparatus. Following are some select, descriptive items regarding the distillation tower construction.

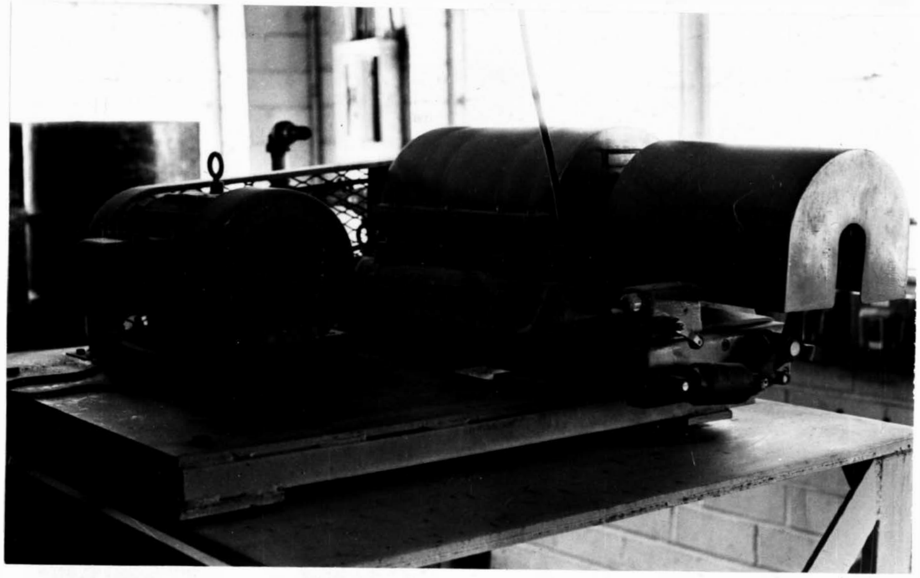


Figure C-4: Centrifuge

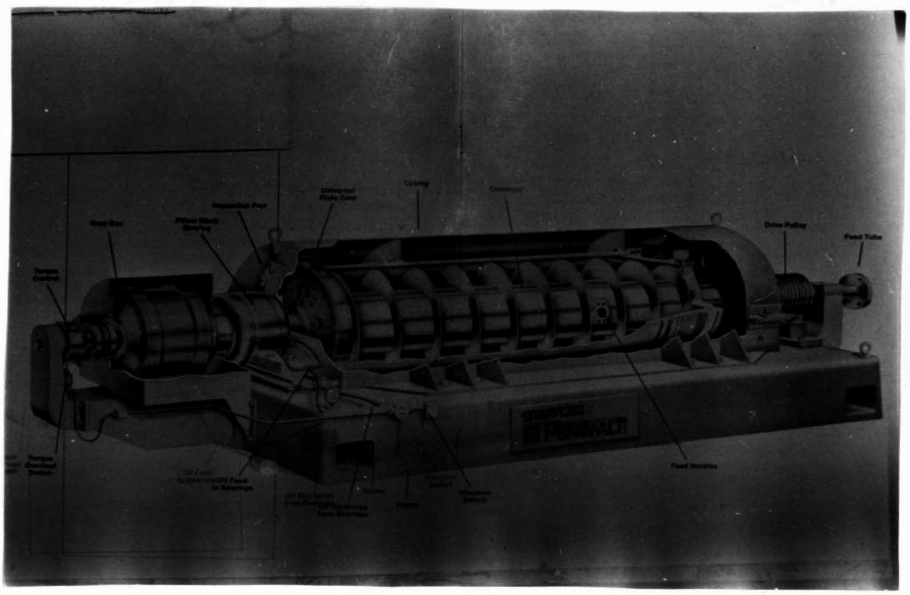


Figure C-5: Centrifuge Construction (Pennwalt Corporation)

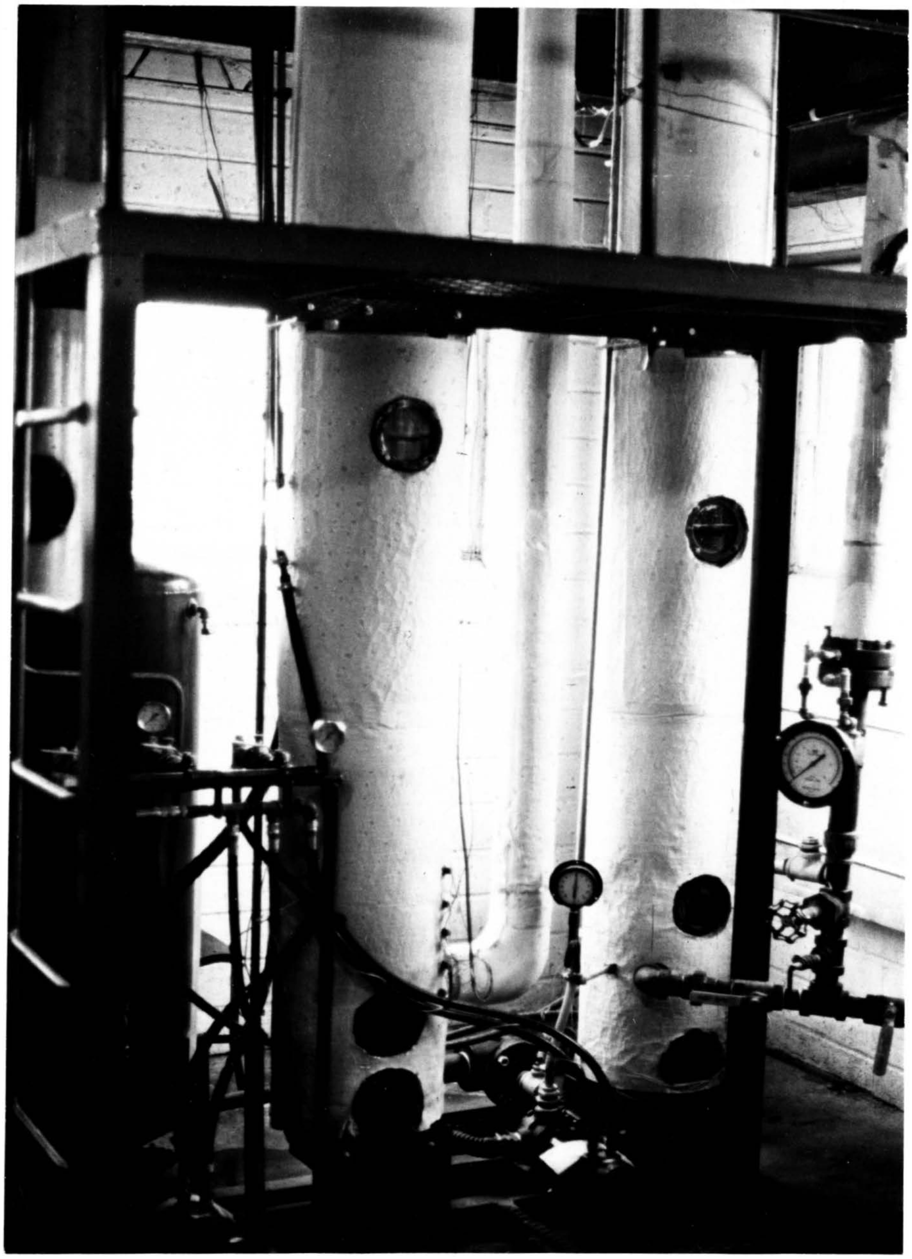


Figure C-6: Distillation Tower

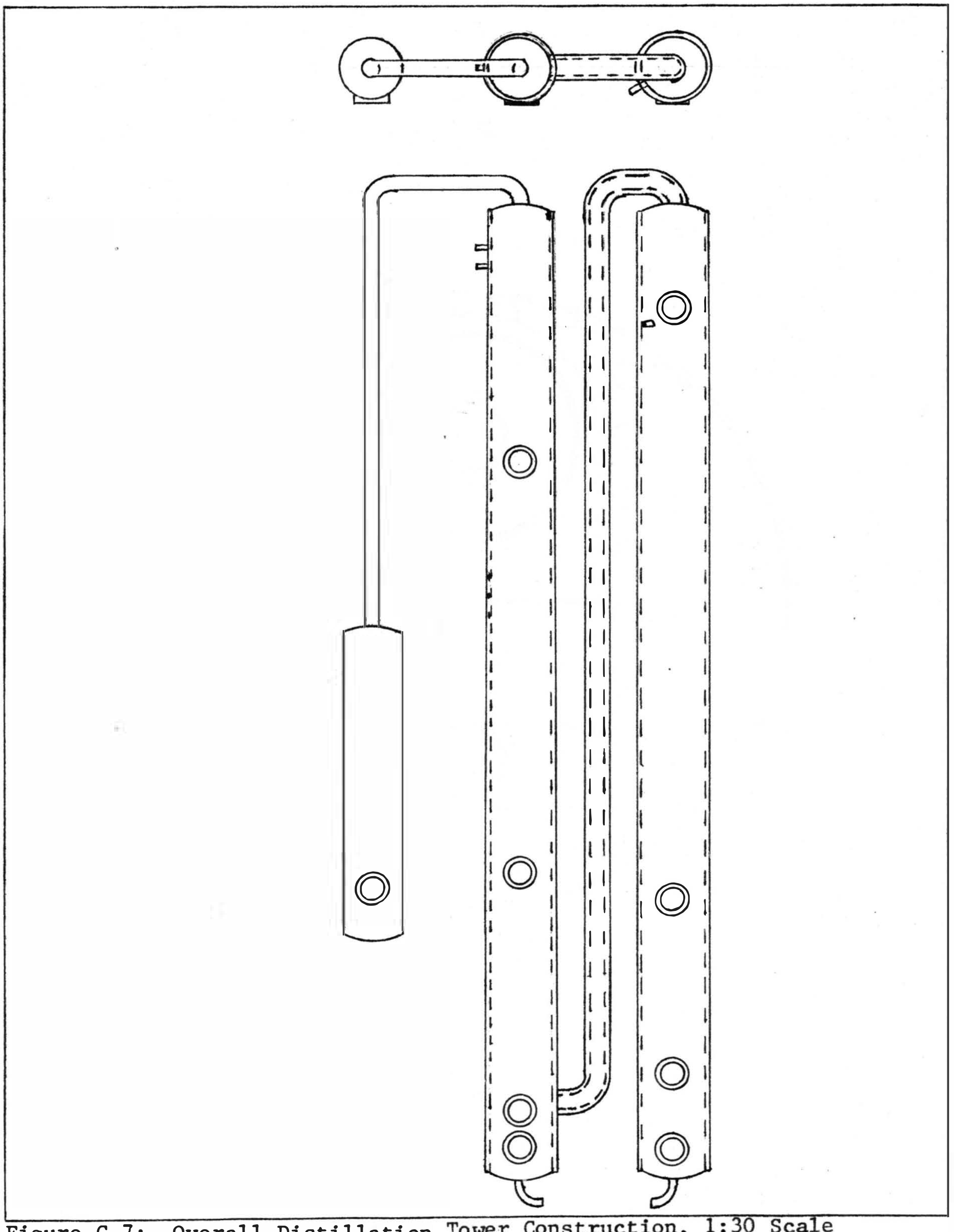


Figure C-7: Overall Distillation Tower Construction, 1:30 Scale

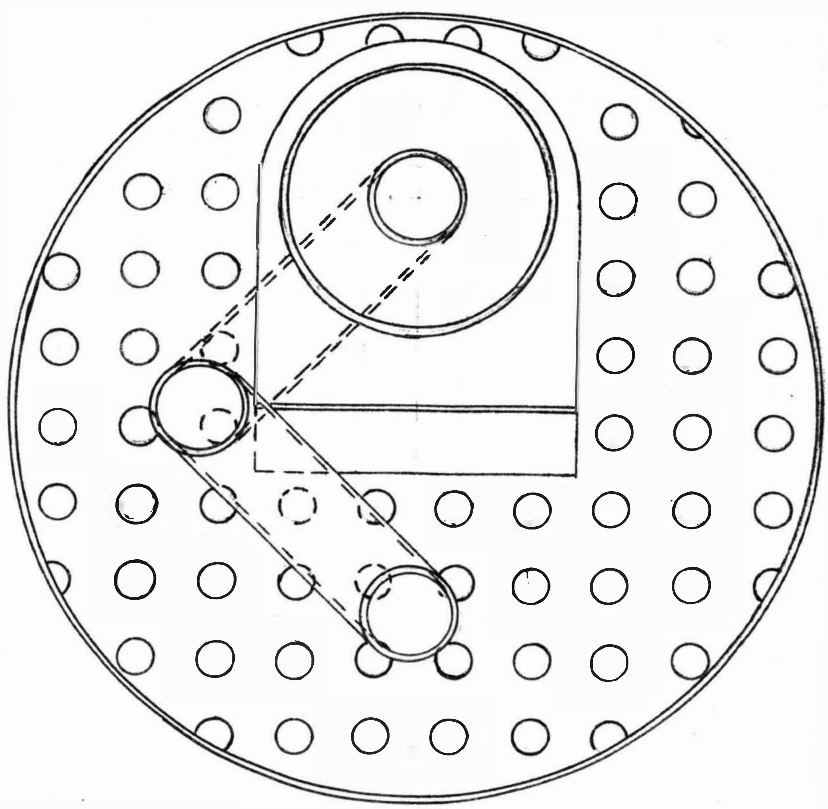


Figure C-8: Top View of Stripper Column, 1:3 Scale

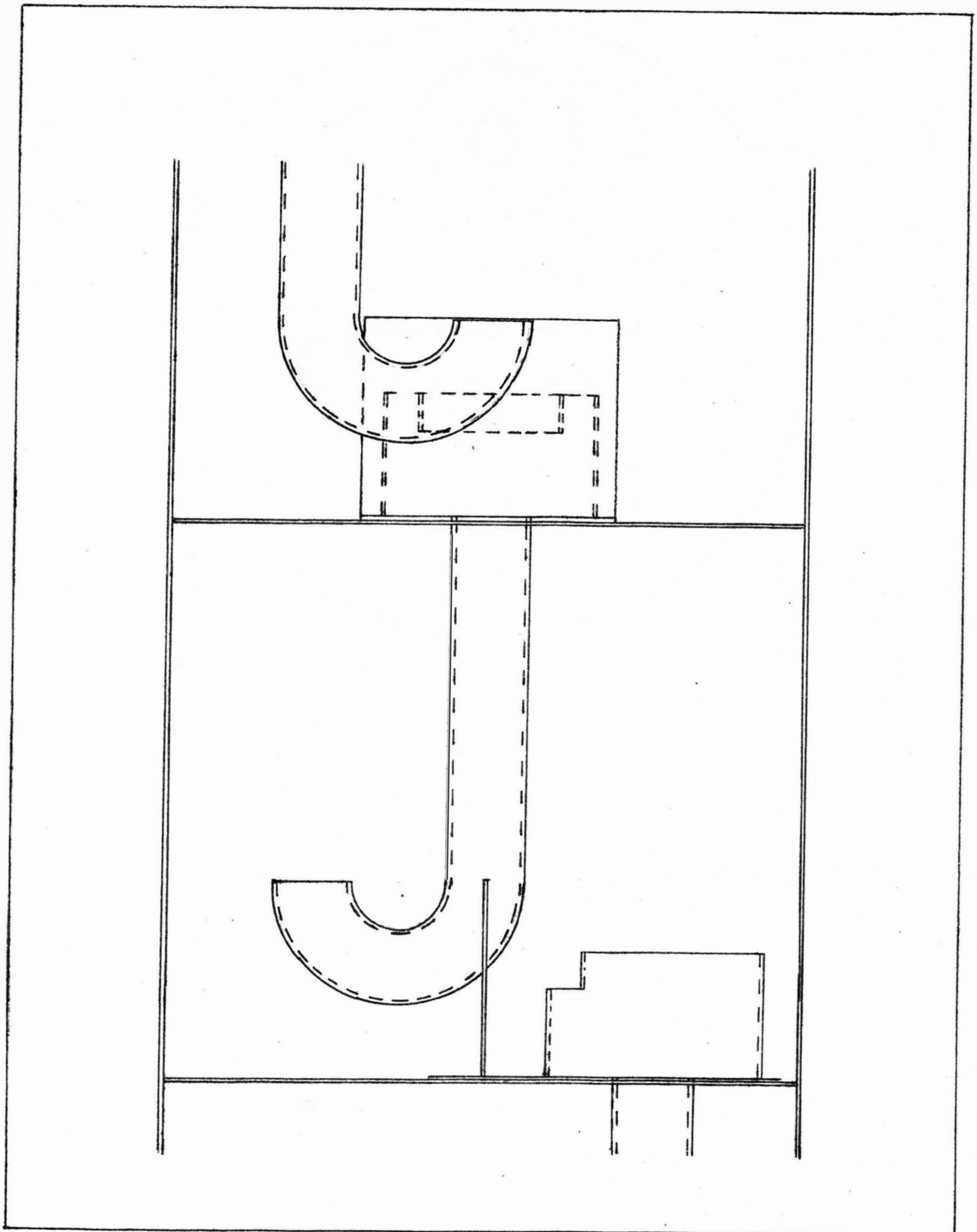


Figure C-9: Side View of Stripper Column Interior, 1:3 Scale

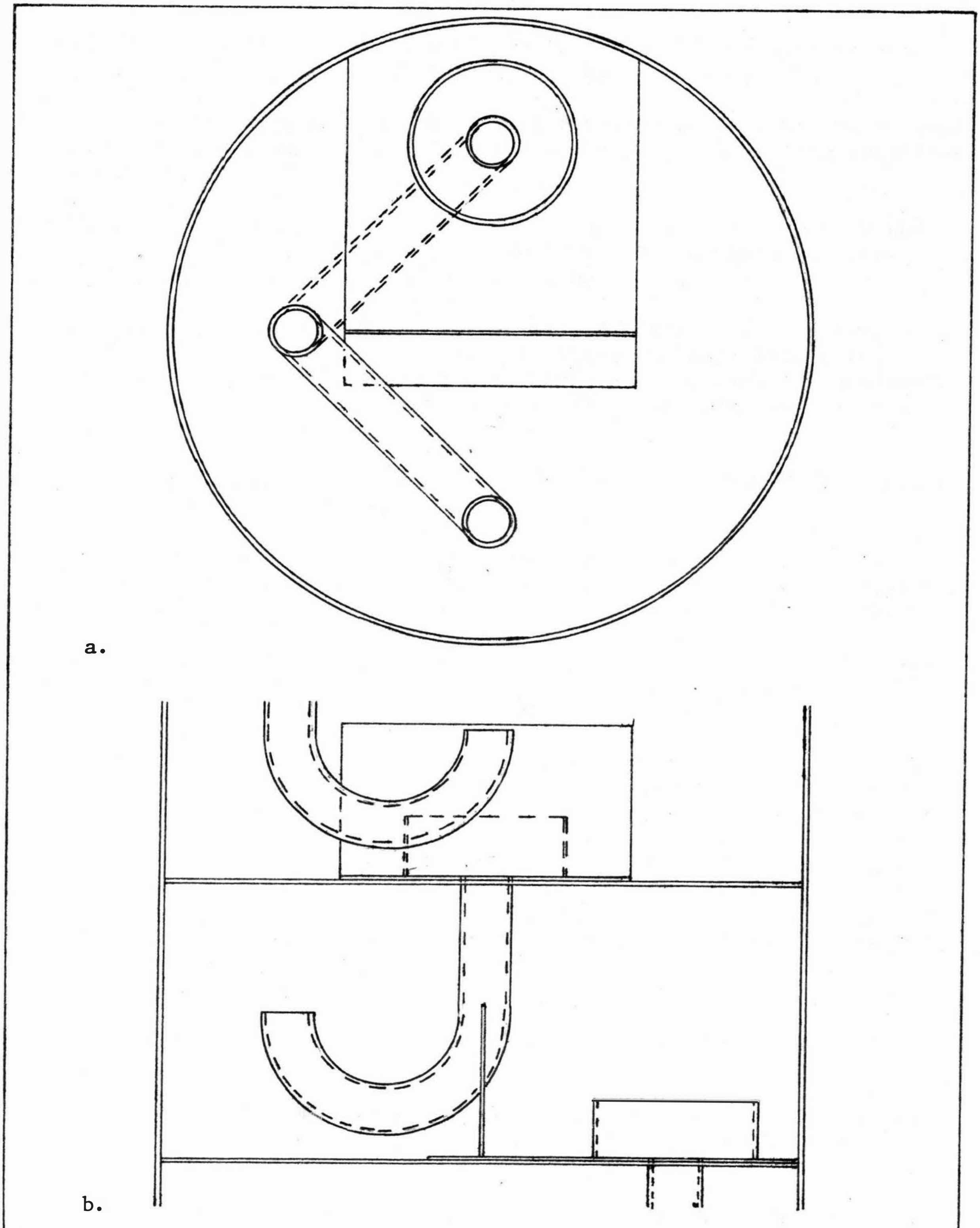


Figure C-10: Top View (a.) and Side View (b.) of Rectifier Column, 1:3 Scale

* A 2.5 centimeter (1.0 inch) shell of fiberglass insulation covers much of the distillation unit (Figure C-7).

* The 7.6 centimeter (3.0 inch) diameter tubing connecting the columns allows for passage of vapor from the stripper column to the rectifier column (Figure C-7).

* The condenser is 152 centimeters (60 inches) tall (Figure C-7) and contains 76.2 linear meters (250 feet) of 1.9 centimeter (three-fourths inch) diameter stainless steel coil.

* The stripper column contains 15 perforated trays (friction fit) at 27.9 centimeter (11.0 inch) intervals below the feed inlet; two above the feed inlet. There are 59 perforations, each 1.2 centimeters (0.50 inch) in diameter, in each tray. This provides an effective open area of 10.2 percent.

* Each J-tube in the stripper column (Figure C-8) is 3.8 centimeters (1.5 inches) in diameter.

* The 7.6 centimeter (3.0 inch) diameter cup (Figure C-8) has a slot cut from it such that a 4.4 centimeter (1.7 inch) liquid level is maintained on each tray in the stripper column during oepration.

* A 12.4 centimeter (4.9 inch) wide by 9.9 centimeter (3.9 inch) tall solid place forces fluid in a circular pattern around the edges of each tray in the stripper column during operation (Figure C-8).

* The rectifier column contains 23 perforated trays (friction fit) at 14.0 centimeter (5.5 inch) intervals in the lower three-fourths of the column. There are 940 perforations (not shown in Figure C-10), each 0.318 centimeter (0.125 inch) in diameter, in each tray. This provides an effective open area of 10.2 percent. A 1.9 centimeter (three-fourth inch) outside diameter and 76.2 meters (250 feet) long stainless steel coil occupies the upper one-fourth of the rectifier column. The purpose of the coil is to use heat from the condensation of reflux in the rectifier column to preheat the corn-beer feed to the stripper column.

* Each J-tube in the rectifier column (Figure C-10) is 2.5 centimeters (1.0 inch) in diameter.

* The 7.6 centimeter (3.0 inch) diameter cup (Figure C-10) has a slot cut from it such that a 3.0 centimeter (1.2 inch) liquid level is maintained on each tray in the rectifier column.

* A 14.0 centimeter (5.5 inch) wide by 7.6 centimeter (3.0 inch) tall plate forces fluid in a circular pattern around the edges of each tray in the rectifier column during operation (Figure C-10).

APPENDIX D

HYDROMETER CALIBRATION

A simple, inexpensive hydrometer is employed to determine the percent by volume of ethyl alcohol in the hydrous (containing water) alcohol product. The temperature-compensated hydrometer is a Tralle & Proof Thermo-Hydrometer, U.S. Custom House Standard, 380 mm , number 11-595 and is available from Fisher Scientific Company, 711 Forbes Avenue, Pittsburgh, PA. 15219. See Figure D-1 for a photograph.

Prior to the use of the hydrometer, calibration tests are performed. Three alcohol samples are used to construct a calibration curve, Figure D-2. The true percent by volume of ethyl alcohol in each aqueous solution is determined by an alternate method of measuring apparent specific gravity. This method is more accurate than the use of the hydrometer, but is much more time consuming. The procedure outlined in Appendix G; 'Percent Ethanol Measurement and Calibration', has produced the results in Table D-1.

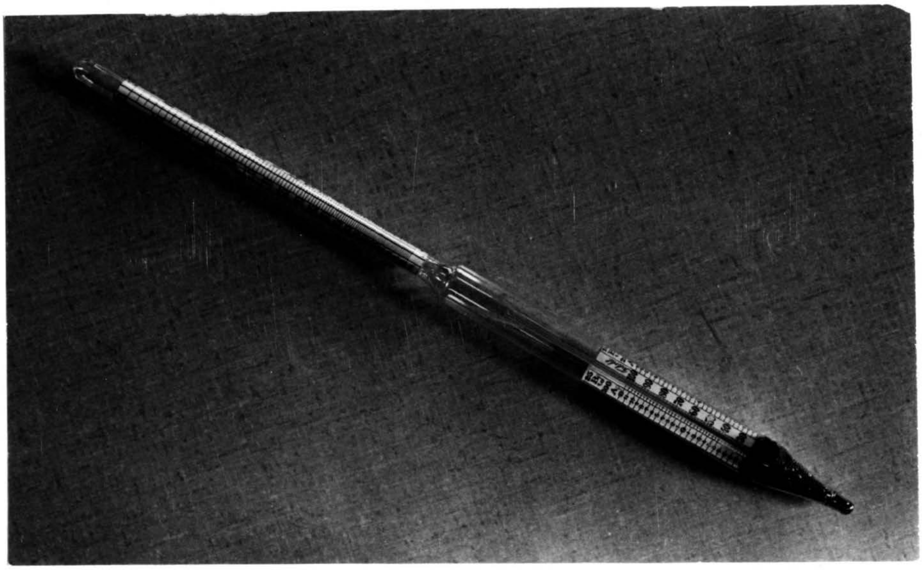


Figure D-1: Temperature Compensated Hydrometer

Table D-1: Hydrometer Calibration Samples. percentages are by volume

	Percent by Volume		
	TEST 1	TEST 2	AVERAGE
SAMPLE 1	93.96	94.01	93.97
SAMPLE 2	91.84	91.78	91.81
SAMPLE 3	89.53	89.53	89.53

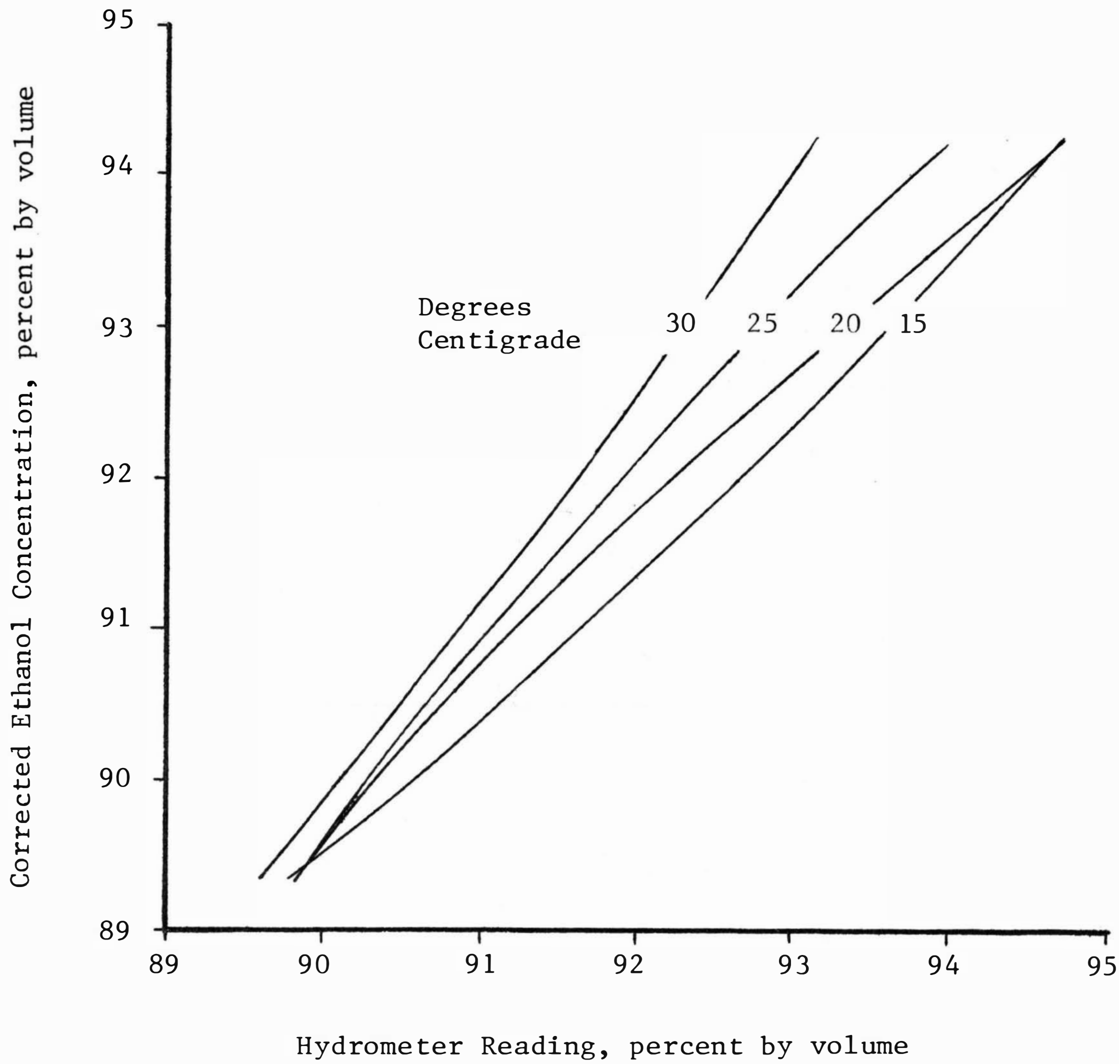


Figure D-2: Hydrometer Calibration Curve

The hydrometer to be calibrated is placed in each sample at various temperatures between 15 and 32°C (60 and 90°F). The technique employed when reading the hydrometer is discussed later. The data thus generated (see Table D-2, below) are employed to construct the calibration curve, Figure D-2.

Table D-2: Calibration Curve Construction Data, percent by volume

SAMPLE 1	SAMPLE 2	SAMPLE 3
94.50 @ 16°C	90.00 @ 18°C	92.50 @ 16°C
94.50 @ 21°C	90.00 @ 20°C	92.00 @ 21°C
93.75 @ 27°C	90.00 @ 26°C	91.75 @ 26°C
93.00 @ 36°C	89.75 @ 33°C	91.50 @ 36°C

Hydrometer Use

The following technique is employed for use of the hydrometer. A 500 millimeter (0.13 gallon), plastic graduated cylinder is employed for taking the sample. Hydrous alcohol is obtained from the distillation tower condenser from between 15 and 32°C (60 and 90°F).

The hydrometer is placed in the sample and allowed to come to thermal equilibrium with the fluid, requiring approximately five minutes. The reading should be taken such that the bottom of the meniscus determines the percent by volume of ethanol in the solution. A temperature compensation is then applied when the sample is at a temperature other than 15°C (60°F). Refer to Figure D-1. Then one uses this temperature compensated value as the absisssa (horizontal axis) of the instrument calibration curve of Figure D-2. The ordinate (vertical axis) then provides a reading accurate to approximately $\pm$ 0.5 percent by volume of ethyl alcohol in the aqueous solution. This reading is contingent upon the assumption that any impurities present have an insignificant effect upon the measurement.

Impurities

Glycerol, succinate, fusel oil, and other impurities have long been known to be by-products of ethanol fermentation; Pyke (1965). Reed and Peppler (1973), and Yang et al. (1982). The formation and relative amounts of the major by-products will be briefly discussed.

Glycerol and Succinate

The production of substantial amounts of glycerol and succinic acid in the fermentation process has long been observed; Pyke (1965), Oura (1977). Its production is influenced by the sugar concentration, fermentation temperature, pH, yeast strain, the amount of oxygen, the sodium chloride concentration, and the sulfide level, according to Yang et al. (1982). It is generally observed that approximately three percent of fermentable sugar is converted to glycerol. Pyke (1965) calculates that succinate consumes another 0.6 percent. Glycerol concentrations in corn-beer are reported by Pyke (1965), Oura (1977), and Yang et al. (1982) to be between 0.11 and 0.25 percent by weight. Oura (1977) has determined that succinate concentrations are approximately one-fifth that amount.

However, very little glycerol or succinate should find its way from the fermented beer to the final product. Like the corn oil found in the corn-beer, the boiling point of glycerol and succinate is substantially above 100°C (212°F). Glycerol boils at 290°C (554°F), succinic acid at 235°C (455°F), (Weast, 1980). Therefore, all of these compounds are elminated from the distillation tower with the spent corn-beer (stillage) and should not be present in the final ethanol product.

Fusel Oil

Fusel oils comprise the second largest group of by-products in alcohol fermentation (Pyke, 1965; Oura, 1977; Yang et al., 1982). The most prevalent fusel oils are 1-propanol, iosbutyl alcohol, active amyl alcohol, and isoamyl alcohol (Webb, 1963; Suomalainen, 1971; and Yant et al., 1982). These compounds are found in fermentation beers in combined concentrations of 0.05 to 0.12 percent by weight under anaerobic conditions. According to Webb (1963), aerobic conditions yield higher fusel oil concentrations by a factor of four.

Unfortunately, the boiling points of many aqueous solutions of fusel oil constituents are between 78 and 100°C (172 and 212°F), Webb (1963). These compounds do not simply escape with the stillage, but are essentially trapped within the distillation unit. Although SDSU does not remove these constituents from the unit, a commercial operation will have to provide for their eventual removal as they may accumulate and disrupt the distillation process. During the short, eight hour runs at SDSU, the compounds did not disrupt the process.

Assuming that all the fusel oil in the beer did find its way over the rectifier tower and into the product, the effect on the hydrometer measurement would still be nil. The specific gravity of these higher alcohols are within three percent of that of ethyl alcohol (Webb, 1963). Since their concentration must be less than 0.5 to 1.2

percent in the product, the error imposed by their presence must be less than 0.04 percent (0.03 x 1.2% = 0.04%). It is safe to say that the effects of these impurities are undetectable.

APPENDIX E

TEMPERATURE MEASUREMENT

Effective operation of the distillation apparatus requires extensive knowledge as to the temperature distribution in the system. The temperature at any particular point in the stripper section determines the extent of ethanol separation and thus the need for adjustments in the steam supply. The temperature at any particular point in the rectifier section dictates the amount of cooling that must be supplied in order to obtain a desired concentration of ethanol in the product.

South Dakota State University utilizes a system of thermisters provided by Sierracin/Western Thermister, 354 Via Del Monte, Oceanside, California 92054 (part number 778C1132). Thirty-six sensors are placed in strategic locations throughout the system and are monitored at a single location. A thermister linearizer and digital display unit provides information to 100ths of a degree centigrade. The electronics consists of a RVC8 Thermister Linearizer and is provided by Intersil, Inc., 10710 N. Tantau Ave., Cupertino, California 95014.

Calibrations of each temperature censor have been made at their respective operating temperatures. A 76MM S-80005-B mercury thermometer made by Sargent Welch Scientific Company has been calibrated at 0.0°C and near 100°C by use of an ice bath and boiling water, respectively. Sensor measurements are then compared to those of the thermometer and corrected accordingly. Figures E-1 and E-2 are typical of the calibration curves employed. The corrected temperature readings are accurate to ± 1 degree centigrade.

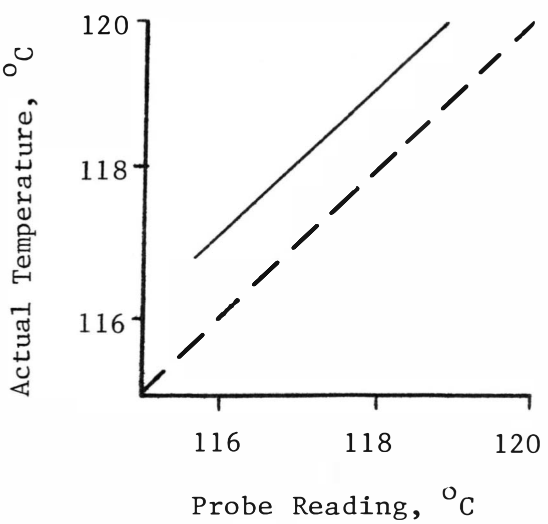


Figure E-1: Steam Temperature Correction

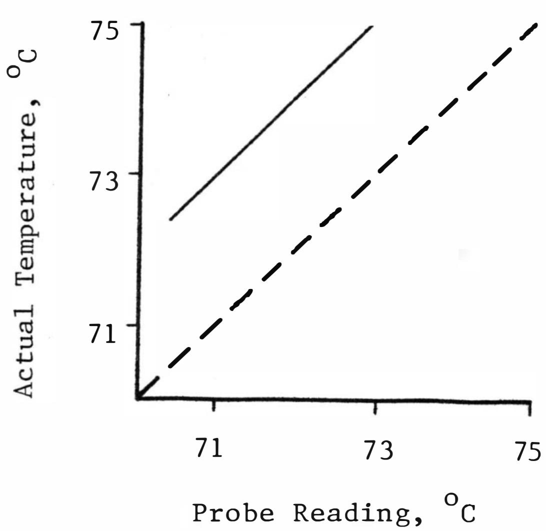


Figure E-2: Corn-Beer Outlet Temperature Correction

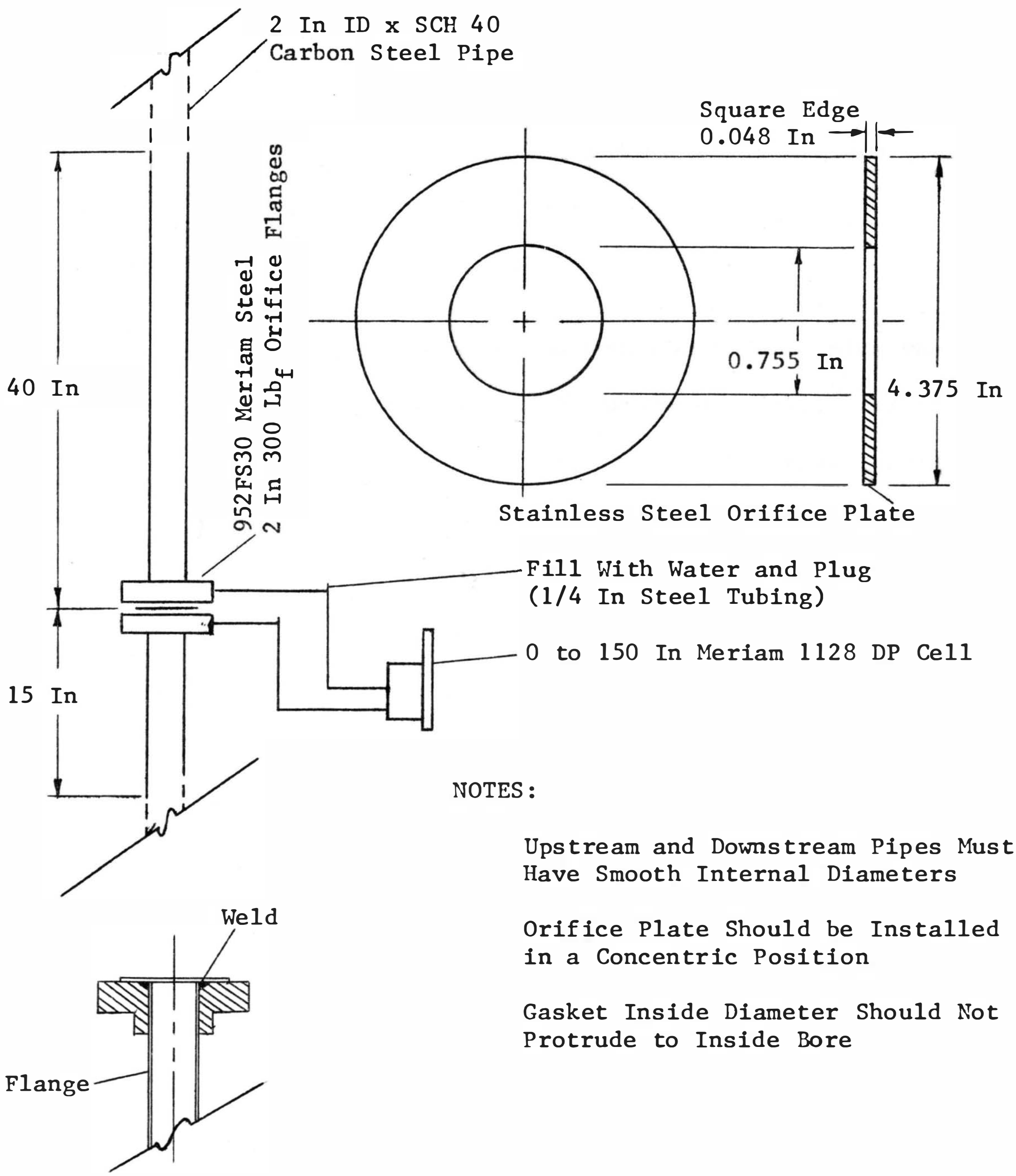
APPENDIX F

STEAM MEASUREMENT

The measurement of steam flow at the SDSU alcohol fuel pilot plant is described herein. Live, saturated steam is directly injected into the 30.5 centimeter (12 inch) diameter, stainless steel stripper section of the distillation unit. Knowledge of the rate of steam flow is employed in the body of this thesis to determine the quantity of energy required to distill ethanol from a corn-water-ethanol slurry.

A set of Meriam Instruments, 952FS30, forged steel orifice flanges are installed in a 5.1 centimeter (2.0 inch) diameter segment of the incoming steam line. Pressure fluctuations measured with a 1126 Meriam Instruments differential pressure (DP) cell were converted to kilograms per hour (pounds mass per hour) steam flow.

Figure F-1 is a drawing of the orifice meter. Specifications on the flanges and DP cell are included. Figure F-2 is a photograph of the steam measurement apparatus.



NOTES:

Upstream and Downstream Pipes Must Have Smooth Internal Diameters

Orifice Plate Should be Installed in a Concentric Position

Gasket Inside Diameter Should Not Protrude to Inside Bore

Figure F-1: Installation of Orifice Meter

The tables and graphs employed in these emperical relationships are in Figures F-3 through F-5 and Tables F-1 through F-3.

The uncertanty in the flow measurement is discussed thereafter. Actual calibration data are then presented to verify the empirical predictions at a single operating point.

Flow Calculations

The following relationship was employed to determine the steam flow rate to the distillation column given a particular DP reading and steam line pressure (Spink, 1972). The relationship and related figures and tables are in the English system of measure.

The final flow rate was converted back to the desired system of measure after the relationship has been utilized.

$$W_h = 359\ S\ D^2\ F_a\ F_m\ F_c\ Y \sqrt{\gamma_f\ h_w}$$

where: W_h is the mass flow rate of the steam in pounds mass per hour.

S is an index for determining d/D, $S = K_1 \beta^2$ where $\beta = d/D$.

D is the inside diameter of the steam pipe in inches.

F_a is the temperature correction factor (orifice expansion factor).

F_c is the viscosity or Reynolds number correction factor.

Y is the gas expansion factor.

γ_f is the steam's specific weight in pounds mass per cubic foot.

h_w is the differential pressure reading in inches of water.

This relationship is valid for compressible gas and is recommended for saturated steam if γ_f is divided by the quality of the steam. Steam quality is defined as the percentage of mass which is in the vaporous phase, typically 95 to 100 percent.

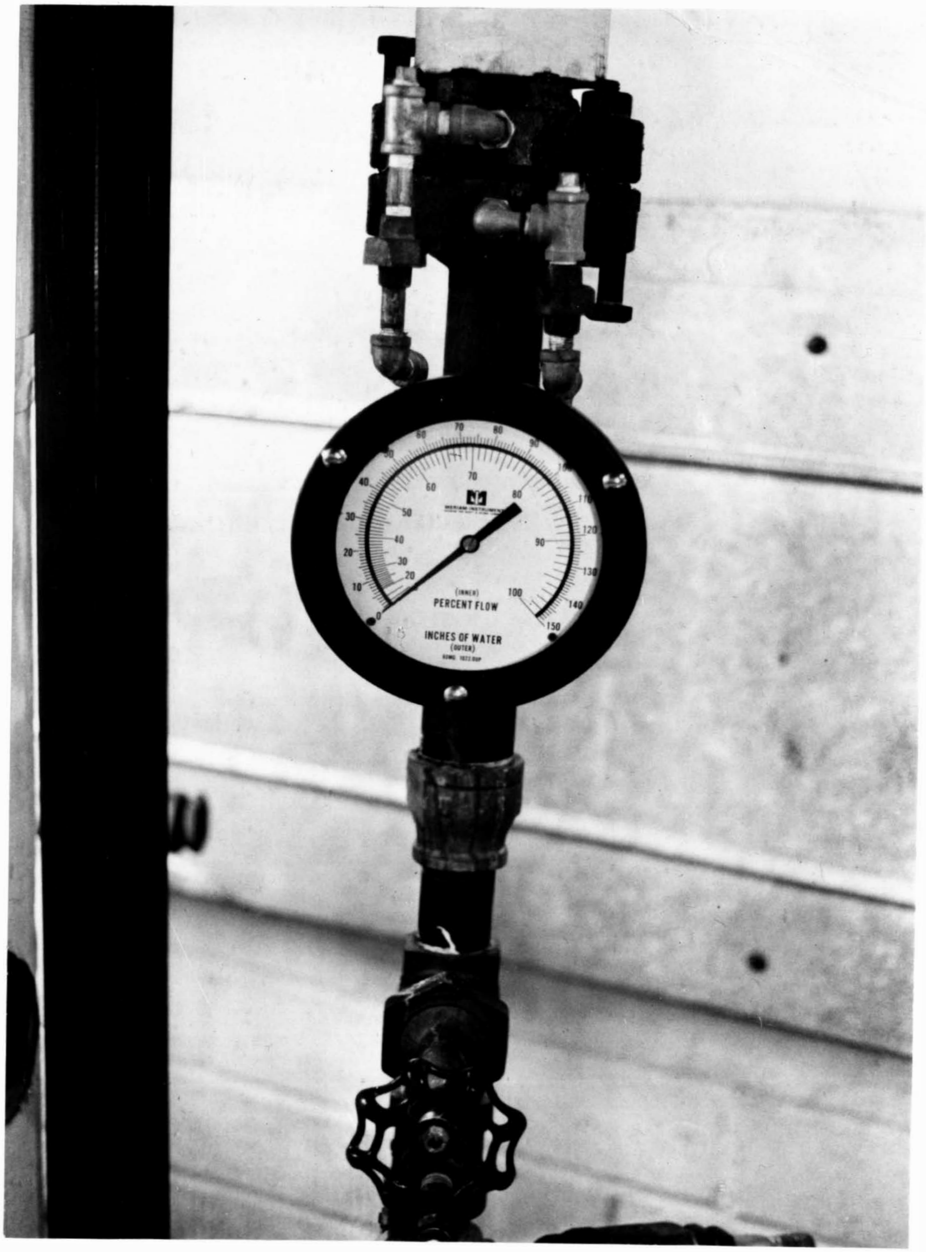


Figure F-2: Steam Flow Measurement Apparatus

Since $S = K_1 \beta^2$; $\beta = d/D$; and, for a diaphragm meter, $F_m = 1$; the relationship can be written in the following manner:

$$W_h = 359\ K_1\ d^2\ F_a\ F_c\ Y\sqrt{\gamma_f h_w}$$

where: W_h is the mass flow rate of the steam in pounds mass per hour.

K_1 is the coefficient of discharge (this factor includes a velocity of approach factor. The operating conditions covered in this paper are slightly out of the range of those recommended by Spink (1972) for determination of K_1. This is taken into account when the uncertainties are examined).

d is the inside diameter of the orifice plate in inches.

F_a is the temperature correction factor (orifice expansion factor).

F_c is the viscosity or Reynolds Number correction factor.

Y is the gas expansion factor.

γ_f is the steam's specific weight in pounds mass per cubic foot (operating conditions at the SDSU alcohol plant are saturated steam at 15 pounds per square inch, gauge. Since the steam line to the still is relatively short; is well insulated; and the steam is throttled from 90 pounds per square inch, gauge; it is assumed that the steam quality is 98 percent. The uncertainty examined later will take into account a quality down to 96 percent).

h_w is the differential pressure reading in inches of water.

For 100 percent gauge flow:

The Reynolds Number is determined as follows:

R_D* is the mass flow rate of the steam in pounds mass per hour.

D is the inside diameter of the steam pipe in inches.

u is the absolute viscosity of the steam in centipoises.

W_h = 340 (assumed)

D = 2.014 (measured)

u = 0.0135 (from Figure F-3)

so R_D = (6.32) X (340) / ((2.014) X (0.0135))

= 79,000

K_1	$= 0.6083$	($\pm$ 0.001)	(from Table F-1)
d	$= 0.755$	($\pm$ 0.002)	(measured)
F_a	$= 1.002$	(0.0005)	(from Table F-2)
F_c	$= 0.995$	(0.002)	(from Figure F-4)
Y^*	$= 0.942$	(0.002)	(from Figure F-5)
$\gamma_f{}^*$	$= 0.0603$	(0.0012)	(from Table F-3)
h_w	$= 150$	(3)	(measured)

so $W_h = 348.7\ lb_m/hr$

Error Analysis

The uncertainty in W_h is found by evaluating the differential of $W_h = 359\ K_1\ d^2\ F_a\ F_c\ Y\sqrt{\gamma_f\ h_w}$ in the following manner:

$$\ln W_h = \ln 359 + \ln K_1 + 2\ln d + \ln F_a + \ln F_c + \ln Y + \tfrac{1}{2}\ln \gamma_f + \tfrac{1}{2}\ln h_w$$

$$\frac{dW_h}{W_h} = 0 + \frac{dK_1}{K_1} + \frac{2\,d(d)}{d} + \frac{dF_a}{F_a} + \frac{dF_c}{F_c} + \frac{dY}{Y} + \frac{d\gamma_f}{2\gamma_f} + \frac{dh_w}{2h_w}$$

$$\left(\frac{dW_h}{W_h}\right)(100\%) = \left(\frac{0.001}{0.6083} + \frac{2(0.002)}{0.750} + \frac{0.0005}{1.002} + \frac{0.002}{0.995} + \frac{0.002}{0.942} + \tfrac{1}{2}\frac{0.0012}{0.0603} + \tfrac{1}{2}\frac{3}{150}\right)(100\%) =$$

$$0.16 + 0.53 + 0.05 + 0.20 + 0.21 + 1.0 + 1.0 =$$

$$3.2\%$$

The uncertainty in W_h at full flow, based on emperical relationships, is theoretically 3.2 percent. This is based upon the uncertainties listed above (Physics Department, 1980).

*The parameters with an asterisk must be re-evaluated for other than 150 inches of water or 15 pounds mass per square inch gauge pressure. The uncertainties associated with each quantity are estimated and placed in parenthesis.

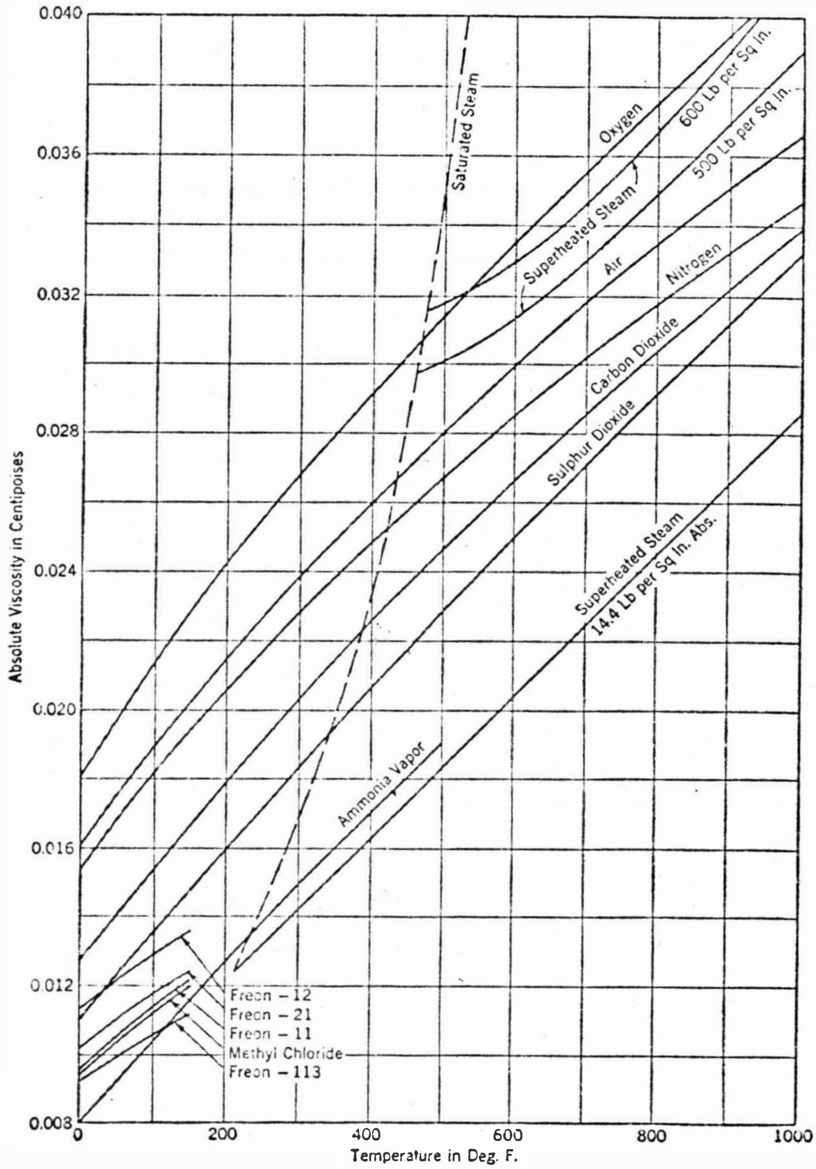


Figure F-3: Absolute Viscosity of Various Gasses (Jennings, 1970)

K_l VALUES FOR FLANGE AND OTHER CLOSE-UP TAPS

$$K_l = 0.598 + 0.01\beta + 0.000001947\,(10\beta)^{4.425}$$

d/D	+.00	+.01	+.02	+.03	+.04	+.05	+.06	+.07	+.08	+.09
.00	-----	.5981	.5982	.5983	.5984	.5985	.5986	.5987	.5988	.5989
.10	.5990	.5991	.5992	.5994	.5995	.5996	.5998	.5999	.6001	.6002
.20	.6004	.6006	.6008	.6011	.6013	.6016	.6019	.6023	.6027	.6031
.30	.6035	.6040	.6045	.6051	.6058	.6065	.6072	.6081	.6090	.6099
.40	.6110	.6121	.6133	.6147	.6161	.6176	.6193	.6210	.6229	.6250
d/D	**+.000**	**+.001**	**+.002**	**+.003**	**+.004**	**+.005**	**+.006**	**+.007**	**+.008**	**+.009**
.500	.6271	.6273	.6276	.6278	.6280	.6283	.6285	.6287	.6290	.6292
.510	.6294	.6297	.6299	.6301	.6304	.6306	.6309	.6311	.6314	.6316
.520	.6319	.6321	.6324	.6327	.6329	.6332	.6334	.6337	.6340	.6342
.530	.6345	.6348	.6351	.6353	.6356	.6359	.6362	.6364	.6367	.6370
.540	.6373	.6376	.6379	.6382	.6385	.6388	.6391	.6394	.6397	.6400
.550	.6403	.6406	.6409	.6412	.6415	.6418	.6421	.6425	.6428	.6431
.560	.6434	.6437	.6441	.6444	.6447	.6451	.6454	.6457	.6461	.6464
.570	.6468	.6471	.6475	.6478	.6482	.6485	.6489	.6492	.6496	.6499
.580	.6503	.6507	.6510	.6514	.6518	.6522	.6525	.6529	.6533	.6537
.590	.6541	.6545	.6548	.6552	.6556	.6560	.6564	.6568	.6572	.6576
.600	.6580	.6584	.6589	.6593	.6597	.6601	.6605	.6610	.6614	.6618
.610	.6622	.6627	.6631	.6635	.6640	.6644	.6649	.6653	.6658	.6662
.620	.6667	.6671	.6676	.6681	.6685	.6690	.6695	.6699	.6704	.6709
.630	.6714	.6718	.6723	.6728	.6733	.6738	.6743	.6748	.6753	.6758
.640	.6763	.6768	.6773	.6778	.6783	.6789	.6794	.6799	.6804	.6810
.650	.6815	.6820	.6826	.6831	.6837	.6842	.6848	.6853	.6859	.6864
.660	.6870	.6875	.6881	.6887	.6893	.6898	.6904	.6910	.6916	.6922
.670	.6928	.6933	.6939	.6945	.6951	.6957	.6964	.6970	.6976	.6982
.680	.6988	.6994	.7001	.7007	.7013	.7020	.7026	.7032	.7039	.7045
.690	.7052	.7058	.7065	.7072	.7078	.7085	.7092	.7098	.7105	.7112
.700	.7119	.7126	.7133	.7140	.7147	.7154	.7161	.7168	.7175	.7182
.710	.7189	.7196	.7204	.7211	.7218	.7226	.7233	.7240	.7248	.7255
.720	.7263	.7270	.7278	.7286	.7293	.7301	.7309	.7316	.7324	.7332
.730	.7340	.7348	.7356	.7364	.7372	.7380	.7388	.7396	.7404	.7413
.740	.7421	.7429	.7437	.7446	.7454	.7463	.7471	.7480	.7488	.7497
.750	.7505	.7514	.7523	.7532	.7540	.7549	.7558	.7567	.7576	.7585

Table F-1: Orifice Expansion Factors for Flange and Other Close-up Taps (Spink, 1972)

F_a VALUES, CORRECTION FOR THERMAL EXPANSION OF PRIMARY DEVICE *

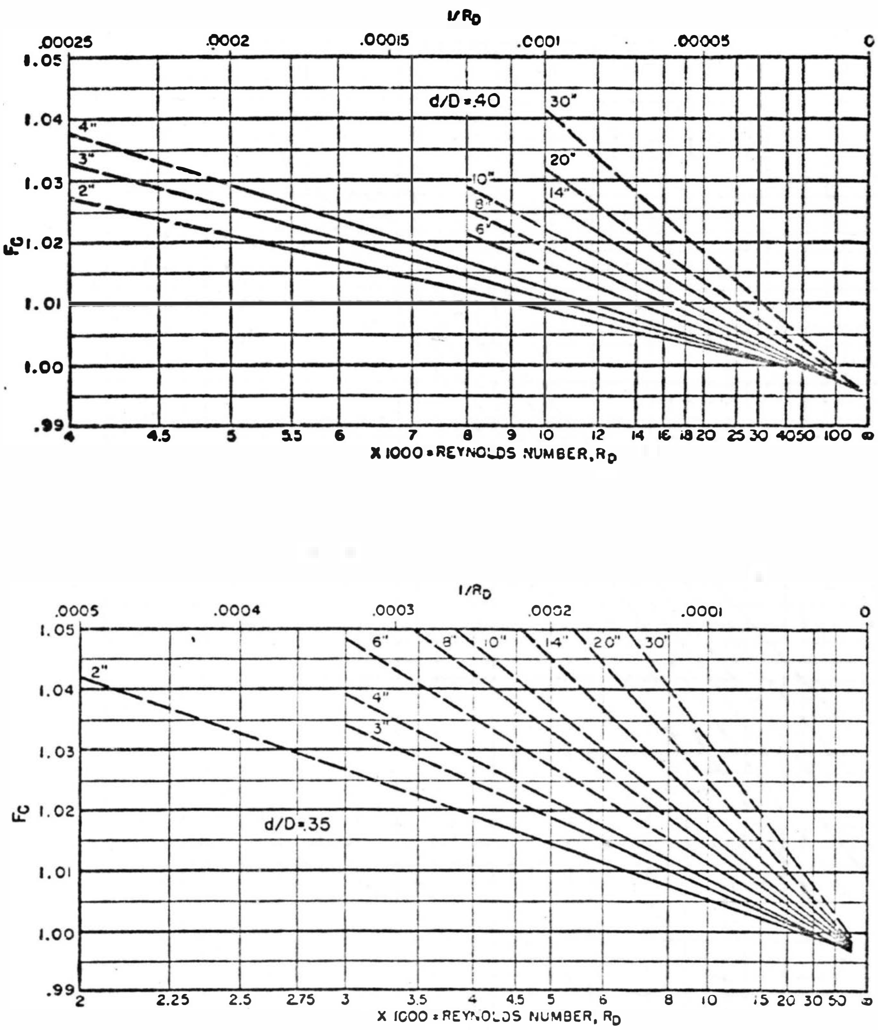
Temperature at Orifice, °F.

Alum.	Copper	Type 430	2% CR MO	5% CR MO	Bronze	Steel	Monel	Type 316 or Type 304	Corr. Factor, F_a
−298					−360				.992
−230	−378				−273				.993
−178	−266				−216			−325	.994
−131	−194				−163		−281	−226	.995
−85	−132				−111		−191	−152	.996
−41	−72				−60		−108	−86	.997
+3	−15				−8		−33	−24	.998
+47	+42	+24	+28	+27	+43	+31	+38	+38	.999
+91	+99	+112	+108	+110	+95	+106	+104	+101	1.000
		+202	+184	+191	+149	+181	+168	+158	1.001
		290	260	272	200	254	230	213	1.002
		374	331	352	249	324	290	266	1.003
		456	400	426	297	392	348	318	1.004
		531	468	493	345	457	405	367	1.005
		604	534	560	393	520	461	414	1.006
		677	597	628	440	542	516	463	1.007
		749	660	696		644	571	512	1.008
		817	720	763		705	626	560	1.009
		884	780	827		765	680	607	1.010
		952	842	888		823	733	655	1.011
		1020	899	948		879	785	703	1.012
		1087	956	1009		934	835	750	1.013
		1155	1011	1071		988	886	796	1.014
		1223	1065	1133		1041	935	840	1.015

* Use one-half the values shown for annular orifices or target meters.

Table F-2: Coefficient of Discharge Factors (Spink, 1972)

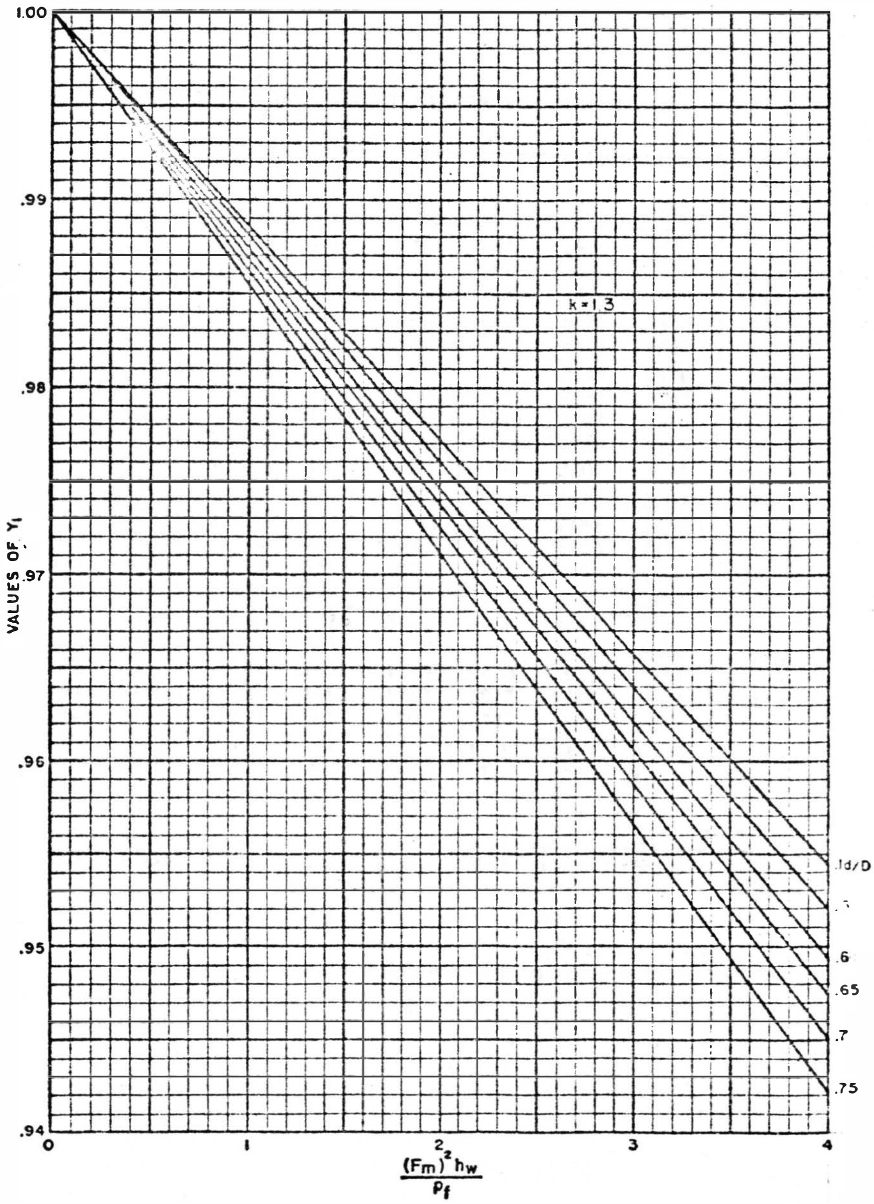
Concentric Orifices, Flange Taps



Reynolds Number Corrections for Concentric Orifices (flange taps)

Figure F-4: Viscosity or Reynolds Number Correction Factors (Spink, 1972)

Y_1 for Natural Gas or Steam, Using Close-Up Taps



Y_1 for Flange or Close-Up Taps, $k = 1.3$

Figure F-5: Gas Expansion Factors (Spink, 1972)

$\sqrt{\gamma_f}$ — SPECIFIC WEIGHT FACTOR FOR DRY STEAM

Temp. °F.	Absolute Pressure, psia 14	14.696	16	18	20	22	24	26	Temp. °C.
Sat.	.1888	.1932	.2010	.2124	.2231	.2333	.2430	.2523	
220	.1873	.1919	.2004	—	—	—	—	—	104.4
240	.1844	.1890	.1973	.2095	.2210	.2320	.2426	—	115.6
260	.1817	.1862	.1944	.2063	.2176	.2285	.2388	.2488	126.7
280	.1791	.1835	.1916	.2033	.2145	.2251	.2353	.2451	137.8
300	.1766	.1810	.1889	.2005	.2115	.2219	.2319	.2416	148.9
320	.1742	.1785	.1864	.1978	.2086	.2189	.2287	.2382	160.0
340	.1720	.1762	.1839	.1952	.2058	.2160	.2257	.2350	171.1
360	.1698	.1740	.1816	.1927	.2032	.2132	.2228	.2320	182.2
380	.1677	.1719	.1794	.1903	.2007	.2106	.2201	.2291	193.3
400	.1657	.1698	.1772	.1880	.1983	.2080	.2173	.2263	204.4
420	.1638	.1678	.1752	.1859	.1960	.2056	.2148	.2236	215.6
440	.1619	.1659	.1732	.1837	.1937	.2032	.2123	.2211	226.7
460	.1601	.1641	.1712	.1817	.1916	.2010	.2099	.2186	237.8
480	.1584	.1623	.1694	.1797	.1895	.1988	.2077	.2162	248.9
500	.1567	.1606	.1676	.1778	.1874	.1966	.2054	.2139	260.0
550	.1528	.1565	.1626	.1733	.1827	.1916	.2002	.2084	287.8
600	.1491	.1527	.1594	.1691	.1783	.1870	.1953	.2033	315.6
650	.1457	.1492	.1557	.1652	.1742	.1827	.1908	.1987	343.3
700	.1425	.1460	.1523	.1616	.1703	.1787	.1866	.1943	371.1
750	.1395	.1429	.1491	.1582	.1668	.1749	.1827	.1902	398.9
800	.1367	.1400	.1461	.1550	.1634	.1714	.1790	.1863	426.7
850	.1340	.1373	.1433	.1520	.1602	.1681	.1755	.1827	454.4
900	.1315	.1348	.1406	.1492	.1572	.1649	.1723	.1793	482.2
	.984	1.033	1.125	1.266	1.406	1.547	1.687	1.828	kg/cm²

* These tables, with additions, are taken from the *U.S. Naval Boiler and Turbine Laboratory Report 1-83*, U.S. Naval Base, Philadelphia 12, Pa. Basic source, *Thermodynamic Properties of Steam*, by Keenan and Keyes, published by John Wiley & Sons, New York (24th printing, 1952.)

Table F-3: Specific Weight Factors (Spink, 1972)

Accurate calibration of any meter employed to measure flow of a gas is a difficult task. The following method has proved sufficient for the purposes of the SDSU alcohol project.

Figure F-6 shows the calibration apparatus. Steam, which normally enters the distillation tower, is diverted to a drum containing cold water. The rate of temperature rise in the drum is recorded for use in determining the amount of thermal energy available in the steam.

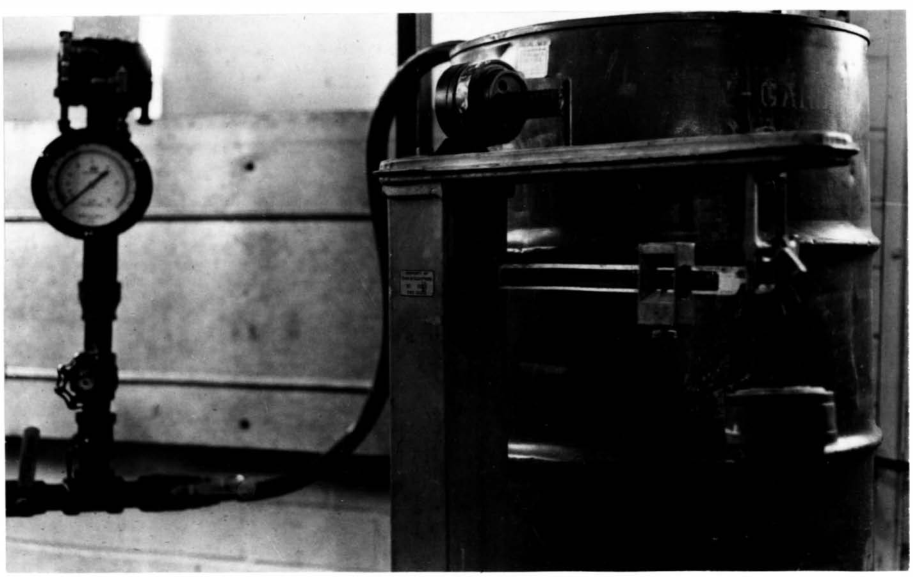


Figure F-6: Steam Meter Calibration Apparatus

Data compiled in this manner are presented in Figure F-7. By assuming a statistically normal distribution, one can employ a 'T' test to arrive at the confidence interval shown. The empirical relationship predicts the flow of energy accurately within the 95 percent confidence limit at this observed point.

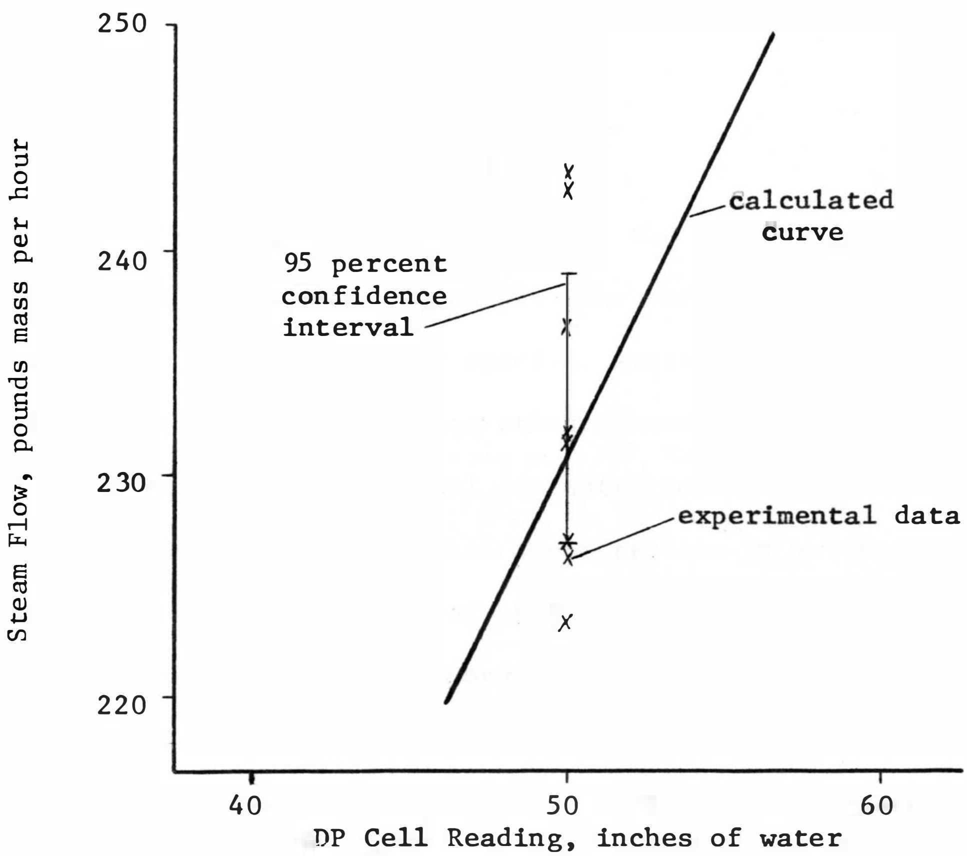


Figure F-7: Confidence Interval for One Point of Steam Meter Operation

APPENDIX G

ETHANOL CONCENTRATION MEASUREMENT

A number of methods for determining the concentration of ethanol in ethanol-corn-water mixtures exist. The method used here involves the measurement of the apparent specific gravity of ethanol-water mixtures obtained from distilling ethanol-corn-water samples. It is employed to determine the ethanol concentration by volume in fermented corn-beer and the residual ethanol concentration by volume in spent stillage. The procedure employed at SDSU, which varies slightly from the method recommended by Horwitz (1970), follows.

A sample of the ethanol-corn-water mixture is taken. To insure representative sampling from the fermenters, the tank's contents are mechanically agitated for at least 15 minutes prior to collection.

One hundred milliliters (0.026 gallon) of the sample is measured into a graduated cylinder and then poured into a spherical, 500 milliliter (0.13 gallon) flask. The cyclinder is rinsed three times with deionized water, the rinsings being added to the flask.

Figure G-1 is a photograph of the distillation apparatus employed to separate the ethanol and water from the corn particles. The sample is boiled slowly for 90 minutes. At this point, virtually all the ethanol and water has been removed from the flask. The flask is cleaned by boiling 200 milliliters (0.053 gallon) of water in it for one hour and scraping with a cleaning brush.

The ethanol-water distillate obtained is then weighed by use of a 200 milliliter (0.053 gallon) pycnometer and compared with the weight of an equal volume of deionized water. (Deionized water yields results identical to those obtained with the distilled water commonly prefered in laboratories.) Before use, the pycnometer is thoroughly cleaned with a chromic acid solution and rinsed three

Figure G-1: Sample Distillation Apparatus times with deionized water. Ten milliliters (0.0026 gallons) of methanol is used for further cleansing. Finally the flask is rinsed three more times with deionized water and considered clean. After being allowed to completely dry, the pycnometer is weighed by use of a Mettler PB300 electronic scale, correct to 0.01 gm.

Next, the pycnometer is filled with deionized water at $20.0 \pm 0.2^{\circ}$ C. The inside of the neck and outside is thoroughly dried with tissue. The weight of the pycnometer and the 200 milliliters (0.053 gallon) of water is observed. The dry weight of the pycnometer is subtracted from this reading and the weight of 100 milliliters (0.026 gallon) of deionized water recorded. The distillate is then placed in the pycnometer and deionized water added to the full mark. After drying again with tissue, the weight of the 200 milliliter (0.053 gallon) pycnometer and its contents are recorded. The weight of the 100 milliliter (0.026 gallon) sample is determined by subtracting the weight of the pycnometer and 100 milliliters (0.026 gallon) of deionized water.

The 100 milliliter (0.026 gallon) ethanol sample weight is divided by the weight of 100 milliliters (0.026 gallon) of deionized water. This yields the apparent specific gravity of the ethanol sample. From specific gravity tables (Horwitz, 1970), one can then readily determine the percent by volume of ethyl alcohol in the corn-beer or stillage being studied.

The error that is associated with this analysis procedure must be determined. The error can be conveniently divided into systematic error and random error.

Systematic Error

Systematic error is consistant error that cannot be considered due to random chance. To characterize the systematic error associated with this procedure, the percentage of ethanol recovered from the sample must be determined. This is done by assuming that the boiling process behaves as a first order system. The concentration of ethanol remaining in the flask is indicated by the overhead vapor temperature and is a function of time.

Figure G-2 has been constructed from the results of boiling two similar samples of corn-beer. First order systems are indicated by the following analysis (Beckwith and Buck, 1969).

First order decay: $T = T_\infty + (T_A - T_\infty)e^{-t/\tau}$ where

- T is the magnitude of any first-order process at $t = t$
- T_∞ is the limiting magnitude of the process as $t \to \infty$
- T_A is the initial magnitude of the process at $t = 0$
- t is time in any appropriate units
- τ is the time constant characteristic of a particular first-order system process

To determine the time constant (τ), three points on the decay curve of Figure G-2 were analyzed. Each time constant as well as the average was calculated. From the figure, $T_\infty = 98.6^oC$ and $T_A = T_{5\ min} = 94.7^oC$

10 minutes: $95.9 = 98.6 + (-4.1)e^{-5/\tau_1}$

$\tau_1 = 12.0$ minutes

25 minutes: $98.0 = 98.6 + (-4.1)e^{-20/\tau_2}$

$\tau_2 = 10.4$ minutes

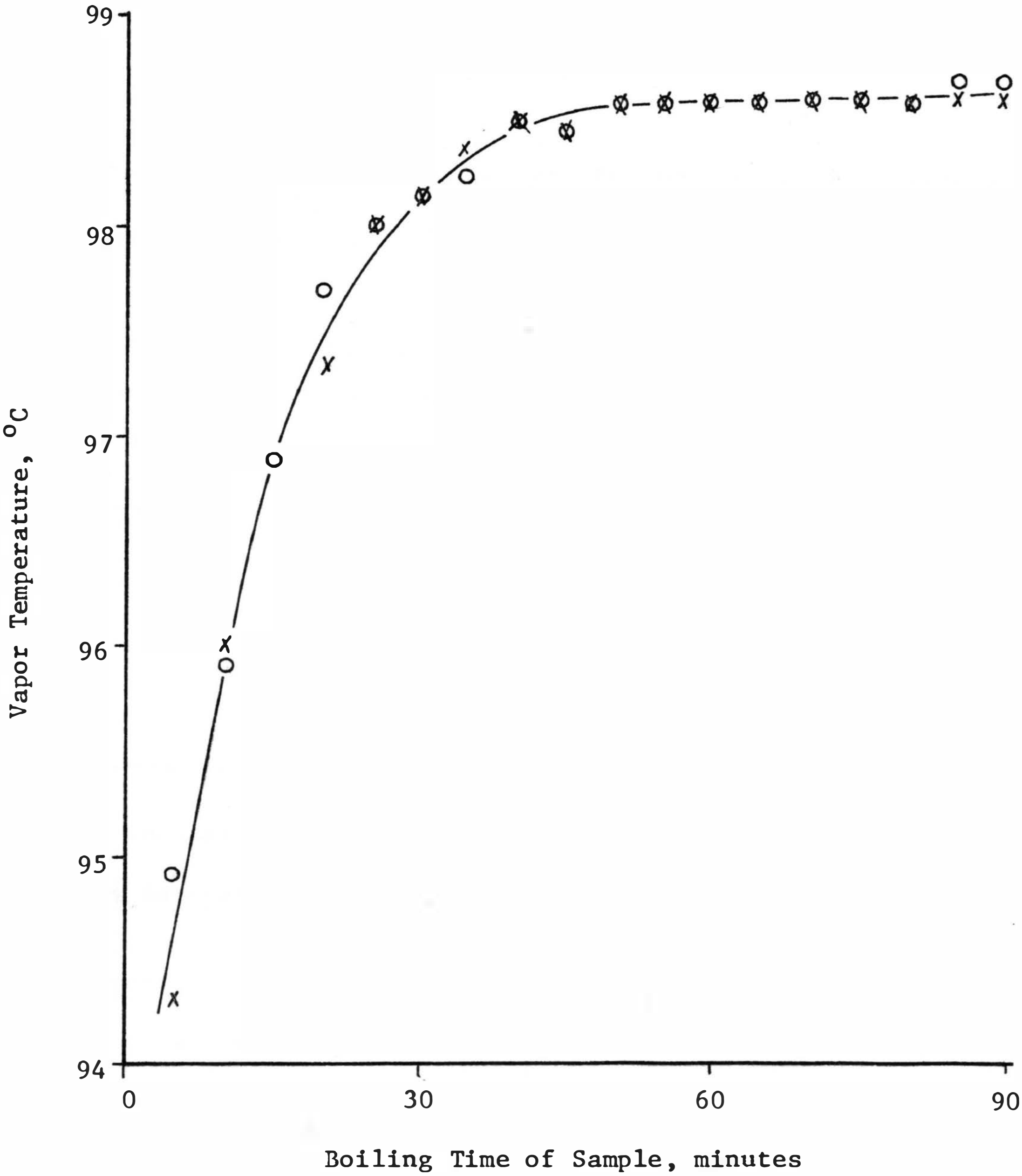


Figure G-2: Evidence of First-Order System of Ethanol-Water Separation

40 minutes: $98.5 = 98.6 + (-4.1)e^{-35/\tau_3}$

$\tau_3 = 9.4$ minutes

$$\tau = \frac{\tau_1 + \tau_2 + \tau_3}{3} = 10.6 \text{ minutes average}$$

Beckwith and Buck (1969) indicate that the properties of a first order system dictate Table G-1.

Table G-1: Time Required to Remove Ethanol in the Boiling Process

Number of Time Constants	Time in Minutes of Boiling Sample	Percentage of Ethanol Removed from sample
1	10.6	63.2
2	21.2	86.5
3	31.8	95.0
4	42.4	98.2
5	53.0	99.3
6	63.6	99.8
7	74.2	99.9
8	84.8	99.9

From this analysis it appears that boiling the sample for ninety minutes is adequate for removal of vertually all the ethanol.

Random Error

The random error associated with this analysis procedure has been determined by testing six samples of stillage, and two sets of six samples of corn-beer. The results of the tests are in Table G-2.

TABLE G-2: Random Error Analysis Data in Percent Ethanol by Volume

	Stillage	Corn-Beer One	Corn-Beer Two
	0.43	8.70	10.71
	0.23	8.64	10.76
	0.40	8.86	11.13
	0.23	8.75	11.15
	0.26	8.75	10.92
	0.30	8.78	10.97
Average:	0.308	8.75	10.94
Standard Deviation:	0.0870	0.074	0.183

The statistical ninety-five percent confidence interval for each set of data follows:

$$\bar{x} - \frac{t_{\alpha/2}s}{\sqrt{n}} < \mu < \bar{x} + \frac{t_{\alpha/2}s}{\sqrt{n}}$$ where

$\bar{x}$ is the sample mean

$t_{\alpha/2}$ is the value of the t distribution, with $v = n - 1$ degrees

s is the sample standard deviation

n is the sample size

μ is the population mean under study

Stillage: $$0.308 - \frac{2.447 \times 0.087}{\sqrt{6}} < \mu < 0.308 + \frac{2.447 \times 0.087}{\sqrt{6}}$$

$$0.22 < \mu < 0.39$$

Corn-Beer: One $$8.75 - \frac{2.447 \times 0.074}{\sqrt{6}} < \mu < 8.75 + \frac{2.447 \times 0.074}{\sqrt{6}}$$

$$8.68 < \mu < 8.82$$

Corn-Beer:
Two

$$10.94 - \frac{2.447 \text{ x } 0.183}{\sqrt{6}} < \mu < 10.94 + \frac{2.447 \text{ x } 0.183}{\sqrt{6}}$$

$$10.76 < \mu < 11.12$$

The accuracy for measuring the ethanol concentration in the corn-beer is adequate for the proposed experimentation. However, based upon these results the procedure is under question when used to determine the residual ethanol concentration in the stillage.